

## Sample Collection Activities for a

## Pilot Study to Investigate the Occurrence of

## Pharmaceuticals and Personal Care Products (PPCPs) in

## Fish Tissue

Contract No. EP-C-04-030

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*Prepared for:*

Office of Water, Office of Science and Technology  
Standards and Health Protection Division  
U.S. Environmental Protection Agency  
1200 Pennsylvania Avenue, NW  
Washington, D.C. 20460

*Prepared by:*

Tetra Tech, Inc.  
400 Red Brook Boulevard, Suite 200  
Owings Mills, MD 21117

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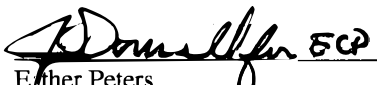
Revision 0

This quality assurance project plan (QAPP) has been prepared according to guidance provided in EPA Requirements for Quality Assurance Project Plans (EPA QA/R-5, EPA/240/B-01/003, U.S. Environmental Protection Agency, Office of Environmental Information, Washington, DC, March 2001) to ensure that environmental and related data are collected, compiled, and/or generated for this project are complete, accurate, and of the type, quantity, and quality required for their intended use. Tetra Tech will conduct work in conformance with the quality assurance program described in the quality management plan for Tetra Tech's Fairfax Group and with the procedures detailed in this QAPP.


## Approvals:

  
Blaine Snyder  
Task Leader  
Tetra Tech, Inc.

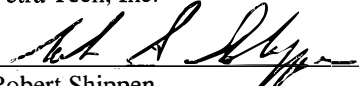
8/2/06  
Date

  
Esther Peters  
Quality Assurance Officer  
Tetra Tech, Inc.


8/3/06  
Date

  
Leanne Stahl  
Project Manager  
U.S. EPA, Office of Science and Technology


8/4/06  
Date

  
Robert Shippen  
Quality Assurance Coordinator  
U.S. EPA, Office of Science and Technology

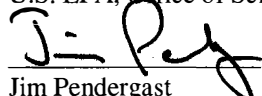
8-16-06  
Date

  
Tangela Cooper  
Project Officer  
U.S. EPA, Office of Science and Technology

8/4/06  
Date

  
Marion Kelly  
Quality Assurance Officer  
U.S. EPA, Office of Science and Technology

8-16-06  
Date

  
Jim Pendergast  
Chief, FSBOD  
U.S. EPA, Office of Science and Technology

8-4-06  
Date

## CONTENTS

Appendices.....	iii
Tables.....	iv
Figures.....	iv
Distribution List.....	v
 A     Project Management	
1.0    Project/Task Organization .....	1
2.0    Problem Definition/Background.....	4
2.1    Research Need .....	4
2.2    Overview of Study Design and Schedule .....	4
3.0    Project/Task Description.....	5
4.0    Quality Objectives and Criteria for Measurement Data .....	7
4.1    Project Quality Objectives .....	7
4.2    Measurement Performance Criteria .....	8
5.0    Special Training Requirements/Certification .....	11
6.0    Documentation and Records .....	11
 B     Data Acquisition	
7.0    Sampling Process Design.....	13
7.1    Sample Type .....	14
7.2    Sampling Period.....	15
7.3    Sample Frame .....	15
7.4    Selection of Sampling Sites .....	15
8.0    Sampling Methods .....	16
8.1    Target Species .....	16
8.2    Composite Sampling.....	16
8.3    Sample Collection.....	17
9.0    Sample Handling and Custody Requirements .....	19
9.1    Sample Handling.....	19
9.2    Sample Integrity.....	20
9.3    Custody Requirements .....	21

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10.0	Analytical Methods Requirements.....	24
11.0	Quality Control Requirements .....	24
12.0	Instrument/Equipment Testing, Inspection, and Maintenance Requirements .....	24
13.0	Instrument Calibration and Frequency.....	25
14.0	Inspection/Acceptance Requirements for Supplies and Consumables .....	25
15.0	Data Acquisition Requirements (Nondirect Measurements) .....	25
16.0	Data Management .....	25
C	Assessment/Oversight	
17.0	Assessment and Response Actions .....	26
18.0	Reports to Management .....	28
D	Data Validation and Usability	
19.0	Data Review, Validation, and Verification Requirements.....	28
20.0	Validation and Verification Methods.....	29
21.0	Reconciliation with Data Quality Objectives .....	29
	Literature Cited .....	30
Appendices		
A	Targeted Wastewater Treatment Plant Sites	
B	Standard Operating Procedure: Fish Tissue Sample Collection Procedures for the PPCP Fish Tissue Pilot Project	
C	Standard Operating Procedure: Flow Measurement Procedures for a PPCP Fish Tissue Pilot Project	
D	Field Data Element Dictionary	
E	Resolved Action Form	
F	Field Sampling Activities Data Review Form	
G	Field Observation Plan	
H	Field Observation Checklist	

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**TABLES**

1	Project Time Line and Milestones .....	6
2	Types of Field Data to Be Collected in Association with Fish Tissue Sample Collection.....	7
3	Recommended Target Species for Inland Freshwaters (in Order of Preference) .....	17
4	Equipment and Supply List for Fish Tissue Sampling .....	19

**FIGURES**

1	Organizational Diagram for the PPCP Fish Tissue Pilot Project.....	2
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**DISTRIBUTION LIST**

Holly Arrigoni  
U.S. EPA Region 5  
77 West Jackson Blvd., WT15J  
Chicago, IL 60604-3507  
312/886-6822 (phone)  
312/886-0168 (fax)

Frank Borsuk  
EPA Region 3/Wheeling Office  
1060 Chapline Street, Suite 303  
Wheeling, WV 26003-2995  
304/234-0241 (phone)  
304/234-0260 (fax)

Tangela Cooper  
USEPA/OST (4305T)  
1200 Pennsylvania Ave., N.W.  
Washington, DC 20460  
202-566-0369 (phone)  
202/566-0409 (fax)

Philip Crocker  
EPA Region 6  
Fountain Place/6WQ-EW  
1445 Ross Avenue, Suite 1200  
Dallas, TX 75202-2733  
214/665-6644 (phone)  
214/665-6689 (fax)

Lonnie Dorn  
EPA Region 4, Science and Ecosystem  
Support Division  
980 College Station Road  
Athens, GA 30605-2720  
706/355-8683 (phone)  
706/355-8726 (fax)

Peter Husby  
EPA Region 9 Laboratory  
1337 S. 46th Street, Building 201  
Richmond, CA 94804-4698  
510/412-2331 (phone)  
510/412-2302 (fax)

Marion Kelly  
USEPA/OST (4303T)  
1200 Pennsylvania Avenue, N.W.  
Washington, DC 20460  
202-566-1045 (phone)  
202/566-1053 (fax)

Jim Pendergast  
USEPA/OST (4305T)  
1200 Pennsylvania Ave., N.W.  
Washington, DC 20460  
202/566-0398 (phone)  
202/566-0409 (fax)

Esther Peters  
Tetra Tech, Inc.  
10306 Easton Place, Suite 340  
Fairfax, VA 22030  
703/385-6000 (phone)  
703/385-6007 (fax)

Jennifer Pitt  
Tetra Tech, Inc.  
400 Red Brook Blvd., Suite 200  
Owings Mills, MD 21117  
410/356-8993 (phone)  
410/356-9005 (fax)

Robert Shippen  
USEPA/OST (4305T)  
1200 Pennsylvania Ave., N.W.  
Washington, DC 20460  
202-566-0391 (phone)  
202/566-0409 (fax)

Blaine Snyder  
Tetra Tech, Inc.  
400 Red Brook Blvd., Suite 200  
Owings Mills, MD 21117  
410/356-8993 (phone)  
410/356-9005 (fax)

Leanne Stahl  
USEPA/OST (4305T)  
1200 Pennsylvania Ave., N.W.  
Washington, DC 20460  
202/566-0404 (phone)  
202/566-0409 (fax)

Michael Walsh  
Computer Sciences Corporation  
6101 Stevenson Avenue  
Alexandria, VA 22304  
703/461-2464 (phone)  
703/461-8056 (fax)

Lynn Walters  
Computer Sciences Corporation  
6101 Stevenson Avenue  
Alexandria, VA 22304  
703/461-2060 (phone)  
703/461-8056 (fax)

## A. PROJECT MANAGEMENT

### 1.0 PROJECT/TASK ORGANIZATION

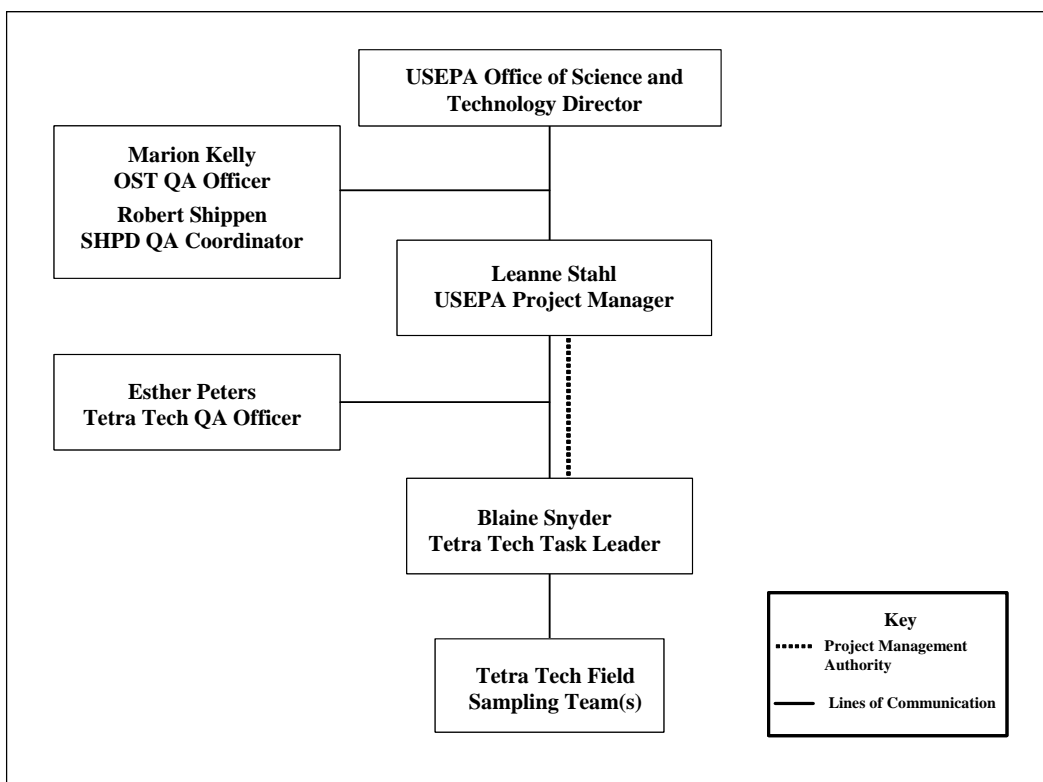
This Quality Assurance Project Plan (QAPP) describes the quality assurance (QA) and quality control (QC) activities/procedures to be used while collecting samples for a pilot study to investigate the occurrence of pharmaceuticals & personal care products (PPCPs) in fish tissue (hereafter referred to as the PPCP Fish Tissue Pilot Project) during the summer and fall of 2006. The purpose of this document is to present the methods and procedures that will be used for the collection of fish tissue from five effluent-dominated streams (in the vicinity of wastewater treatment plant discharges) and one reference stream, and the quality assurance procedures that will be employed. This document addresses *only* the sample collection effort of the PPCP Fish Tissue Pilot Project.

This QAPP was prepared according to guidance presented in the document *EPA Requirements for Quality Assurance Project Plans*, EPA QA/R-5 (USEPA 2001). References to the QAPP elements described in the guidance document are included herein. The sample collection methods, procedures and protocols follow the guidelines and recommendations of *Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories. Volume I: Fish Sampling and Analysis, Third Edition* (USEPA 2000).

The project team organization provides the framework for conducting the sample collection task to meet study objectives. The organizational structure and function also facilitate project performance and adherence to QC procedures and QA requirements. Key roles are filled by those persons responsible for ensuring the collection and processing of valid data and for routinely assessing the data for precision and accuracy, as well as the persons responsible for approving and accepting final products and deliverables. The project and QA personnel include staff from USEPA and Tetra Tech. The project organizational chart is presented in Figure 1, and it includes relationships and lines of communication among key project team members.

The **USEPA Project Manager** is Leanne Stahl, who will supervise the assigned project personnel to provide for their efficient utilization by directing their efforts either directly or indirectly. As Project Manager she will also have the following responsibilities:

- providing oversight for study design, site selection, and adherence to design objectives,
- reviewing and approving the project work plan, QAPP, and other materials developed to support the project, and
- coordinating with contractors and reviewers to ensure technical quality and contract adherence.



**Figure 1.** Organizational Diagram for the PPCP Fish Tissue Pilot Project.

The **OST Quality Assurance Officer** is Marion Kelly, who will be responsible for reviewing and approving all Quality Assurance Project Plans (QAPPs). The **SHPD Quality Assurance Coordinator** is Robert Shippen, who will be responsible for reviewing and recommending approval of all QAPPs. Additional OST QA Officer and SHPD QA Coordinator responsibilities include the following:

- reviewing and evaluating field procedures,
- conducting external performance and system audits of the procedures, and
- participating in Agency QA reviews of the study.

The **Tetra Tech Task Leader** is Blaine Snyder, who will participate in study design and site selection processes and supervise planning and implementation of field collection activities. He is responsible for ensuring that QA/QC protocols are maintained throughout the sample collection, handling, and shipment processes. Other specific responsibilities of the Task Leader include the following:

- coordinating project assignments in establishing priorities and scheduling,

- ensuring completion of high-quality projects within established budgets and time schedules,
- providing guidance, technical advice, and performance evaluations to those assigned to the project,
- implementing corrective actions and providing professional advice to staff,
- preparing and/or reviewing preparation of project deliverables,
- providing support to USEPA in interacting with the project team, technical reviewers, and USEPA Regions/States to ensure technical quality requirements are met in accordance with project design objectives,
- supervising field sample collection activities, and
- reviewing all required documentation for completeness, seeing that any problems encountered outside normal operating conditions are documented and addressed, and verifying all other QA/QC procedures identified in the QAPP are followed.

The **Tetra Tech Quality Assurance (QA) Officer** is Esther Peters, whose primary responsibilities include the following:

- monitoring quality control (QC) activities to determine conformance,
- reviewing the QAPP for completeness and noting inconsistencies,
- providing support to USEPA and the Tetra Tech Task Leader in preparation of the work plan and QAPP and in their distribution, and
- approving the QAPP.

**Field Sampling Teams** will be composed of contractor and university-affiliated field staff. The Task Leader will direct and supervise the Field Sampling Teams and provide for their efficient utilization by directing their efforts. Field personnel are responsible for performing all field work, including collection, preparation, and shipment of fish tissue samples and completion of field sampling records. The Field Sampling Teams will be composed of scientific staff with specialization and technical competence in field sampling activities to effectively and efficiently perform the required work. They must perform all work in adherence with the project work plan and QAPP, including maintenance of sample custody and related documentation. Custody procedures are required to ensure the integrity of the samples with respect to prevention of contamination and maintenance of proper sample identification during handling. In this role, Field Sampling Teams are responsible for:

- receiving and inspecting the sample containers,

- completing and signing appropriate field records,
- assigning tracking numbers to each sample,
- verifying the completeness and accuracy of chain-of-custody documentation,
- controlling and monitoring access to samples while in their custody, and
- initiating shipment of the samples to appropriate destinations.

## **2.0 PROBLEM DEFINITION/BACKGROUND**

### **2.1 Research Need**

EPA's Office of Science and Technology (OST) within the Office of Water (OW) is initiating a pilot study to investigate the occurrence of pharmaceutical and personal care product (PPCP) chemicals in fish tissue called the PPCP Fish Tissue Pilot Project. Increasing evidence indicates widespread occurrence of PPCP compounds in surface water, sediments, and municipal effluent, but data on the accumulation of PPCP compounds in fish tissue are scarce. This study was planned to respond to EPA's new priority of obtaining environmental data on emerging contaminants and to increase the data available on the occurrence of PPCP compounds in fish. The proposed targeted study design calls for collecting fish samples from five effluent-dominated streams in the vicinity of wastewater treatment plant (WWTP) discharges and one reference site. Tissue fractions from each fish sample (fillets and livers) will be analyzed for 39 PPCP chemicals. These analyses will provide data to determine the potential of the target chemicals to survive the wastewater treatment process and to bioaccumulate in the tissue of fish.

Tetra Tech, Inc. has been tasked with planning, implementing, and managing sample collection, preparation and analysis, and forwarding pilot project results to Computer Sciences Corporation (CSC) for data review and database development. Tetra Tech will collect all samples, and will subcontract with a laboratory for the preparation and analysis of fish samples. This QAPP describes only the requirements for sample collection activities. The analytical laboratory (to be determined) will be subcontracted to analyze PPCP Fish Tissue Pilot Project samples for the 39 target chemicals using analytical methods, such as high performance liquid chromatography-tandem mass spectrometry (HPLC-MS/MS), gas chromatography/mass spectrometry (GC/MS), or other methods that can achieve the method detection limits specified by EPA. Analytical QA/QC procedures and requirements will be documented in a separate QAPP.

### **2.2 Overview of Study Design and Schedule**

EPA is conducting the PPCP Fish Tissue Pilot Project to investigate the occurrence of a broad suite of PPCPs (39 compounds) in the tissue of adult freshwater fish. These fish will be collected from streams in the vicinity of wastewater treatment plant (WWTP) discharges to determine the potential for the target PPCP compounds to bioaccumulate in fish tissue.

Sampling sites will be selected on five effluent-dominated streams and at one reference site. Appendix A contains a prioritized list of target sampling sites.

Six composite samples of a single fish species will be collected from each stream. Each composite will consist of a minimum of three adult fish. Depending on the size of the fish available at the site, the number of fish in a composite sample may need to be adjusted to provide sufficient tissue for analysis. Sampling is scheduled for the summer and fall of 2006. Whole fish composites will be shipped to the laboratory on dry ice. The laboratory will prepare two tissue fractions from each fish composite sample for analysis: liver tissue to address the occurrence of PPCP compounds in fish, and muscle tissue (fillets) to determine human exposure through fish consumption. The laboratory will complete analyses for one or two sites in September 2006 and deliver the full analytical data set by March 2007.

### 3.0 PROJECT/TASK DESCRIPTION

The study design reflects the study goal and objectives defined by USEPA. The study goal can be stated simply — *to investigate the occurrence of a broad suite of PPCPs (39 compounds) in the tissue of harvestable sized adult freshwater fish that are typically consumed by wildlife and humans*. The project field sampling task presented and discussed in this document involves only those methods and procedures used to collect and ship fish tissue samples for the PPCP Fish Tissue Pilot Project. The Analytical Activities QAPP for the PPCP Fish Tissue Pilot Project discusses the following study topics and tasks: sample preparation, compositing and homogenization; target analytes; analytical methods; and sample analysis.

In consultation with the USEPA Office of Science and Technology, Tetra Tech will coordinate with USEPA headquarters and regional staff to collect fish tissue samples from targeted effluent-dominated streams in the contiguous United States. Field teams will sample five sites where waters are dominated by wastewater treatment plant (WWTP) effluent, along with one reference quality site (Appendix A). The samples will be collected between August and November of 2006. The fish tissue samples will be collected based on a targeted design to provide information on the occurrence of PPCP contaminants in fish. The following elements must be considered when planning field logistics:

- Field teams will consist of one experienced fisheries biologist, who will also serve as the quality control specialist, and two field technicians, all of whom must have experience with the array of fisheries sampling gear types to be used.
- The pilot study will focus on a single resident species (subject to effluent exposure for much of its life cycle) that is typically consumed by wildlife and humans (Section 8.1).
- The six samples from each site must consist of a composite of fish (e.g., a minimum of 3 individuals that will collectively provide at least 30 grams of edible tissue and 10 grams of liver tissue) of the same target species and be the same relative size from each sampling location (Section 8.2).

- Field sampling activities will be conducted from August through November of 2006. All activities associated with fish tissue sample collection will be conducted consistent with the requirements and procedures (Appendix B) specified in this QAPP as approved by the USEPA Project Manager. Sampling activities will conclude with the development of a field collection effort report (i.e., detailed listing of all sampling participants, sampling locations, and specimens collected), and a Field Sampling QA Report by Tetra Tech which will be reviewed by the USEPA Project Manager. The summary will be used to document and report back to USEPA regarding the collective sampling activities. Implementation of the field sampling task will proceed with several milestones, as presented in Table 1.

<b>Activities and Milestones (2006 - 2007)</b>	<b>2006</b>												<b>2007</b>		
	J	F	M	A	M	J	J	A	S	O	N	D	J	F	M
Literature search for background information and current research															
Develop study design and solicit labs for sample analysis															
Develop QAPP and project sampling plan for field efforts															
Sampling of streams and shipment of samples to analytical laboratory															
Tissue analysis (refer to Analytical Activities QAPP)															
Data review and validation (refer to Analytical Activities QAPP)															
Interim results (refer to Analytical Activities QAPP)															
Full analytical data set (refer to Analytical Activities QAPP)															

## 4.0 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

### 4.1 Project Quality Objectives

Data of known and documented quality are essential to the success of any monitoring or sampling program. Data quality objectives (DQOs) are qualitative and quantitative statements that clarify the intended use of the data, define the type of data needed to support the decision, identify the conditions under which the data should be collected, and specify tolerable limits on the probability of making a decision error due to uncertainty in the data. DQOs are developed by data users to specify the data quality needed to support specific decisions. Sources of error or uncertainty include the following:

- Sampling error: The difference between sample values and *in situ* true values from unknown biases due to collection methods and sampling design,
- Measurement error: The difference between sample values and *in situ* true values associated with the measurement process,
- Natural variation: Natural spatial heterogeneity and temporal variability in population abundance and distribution, and
- Error sources or biases associated with compositing, sample handling, storage, and preservation.

This QAPP addresses only fish tissue sample collection activities, so the relevant quality objectives are related to sample handling issues. One exception involves the measurement of stream flow. Study DQOs for measuring stream flow will require that flow meters are calibrated to a known standard as per manufacturer's specifications (Appendix C). Types of field sampling data needed for this project are listed in Table 2. Discussion of conventional data quality indicators, i.e., precision, accuracy, completeness, representativeness, and comparability, follows in this section. Methods and procedures described in this document are intended to reduce the magnitude of the sources of uncertainty (and their frequency of occurrence) by applying the following approaches:

- use of standardized sample collection and handling procedures, and
- use of trained scientists to perform the sample collection and handling activities.

**Table 2.** Types of Field Data to Be Collected in Association with Fish Tissue Sample Collection.

<b>Data Type</b>	<b>Measurement Endpoint(s) or Units</b>
Fish specimen	Species-level taxonomic identification
Fish Length	Millimeters (mm), total length
Fish Weight	Grams (g)
Estimated Average Stream Depth	Meters (m)
Estimated Average Stream Width	Meters (m)
Stream Flow	Meters/Second (m/sec)

## 4.2 Measurement Performance Criteria

Measurement performance criteria are quantitative statistics that are used to interpret the degree of acceptability or utility of the data to the user. These criteria, also known as data quality indicators (DQIs), include the following:

- precision,
- accuracy,
- representativeness,
- completeness, and
- comparability.

### *Precision*

Precision is a measure of internal method consistency. It is demonstrated by the degree of agreement between individual measurements (or values) of the same property of a sample, measured under similar conditions. As the analytical testing is beyond the scope of this QAPP, no specific criteria are required for this parameter. However, sufficient sample volumes (i.e., at least three fish per composite as described in Section 8.2) will be collected to allow for the assessment of precision during analytical laboratory testing.

For this study, all fish in a stream cannot be sampled, and the laboratory analytical process is not perfect. The combined variability introduced by the sampling at a stream, the compositing of fish, the subsampling of the composite for analysis, and the chemical analysis itself can be considered the “index” variability. The detection limits and analytical precision are one part of the analytical process that can be specified ahead of time (however, analytical processes are not part of this QAPP). The training of sampling crews, the procedures that they use to collect fish from a stream, and the Field Record Forms on which they record data and sampling details will also be standardized. Only two sampling crews will be collecting fish for this study, which will greatly reduce variability. Besides standardizing training and Field Record Forms, this dimension of variability cannot be reduced. Sampling method consistency and adherence to Standard Operating Procedures (Appendix B) will be assessed during field audits and documented in a Field Sampling QA Report. In the event that the field audit identifies problems requiring attention, the Tetra Tech Task Leader and/or the USEPA QA Officer will immediately consult with the USEPA Project Manager. The corrective action system for this project is described in Section 17.0.

### *Accuracy*

Accuracy is defined as the degree of agreement between an observed value and an accepted reference or true value. For example, accuracy of flow meters used for this study will be assured through proper calibration to known standards (Appendix C). Accuracy is a combination of random error (precision) and systematic error (bias), introduced during sampling and analytical operations. Bias is the systematic distortion of a measurement process that causes errors in one direction, so that the expected sample measurement is always greater or lesser to the same degree than the sample’s true value. As mentioned previously, since analytical testing is beyond the

scope of this QAPP, no accuracy criteria are identified here. However, proper sample handling procedures (Section 9.1) will be followed to minimize sample contamination.

### *Representativeness*

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter, variations at a sampling point, a process condition, or an environmental condition.

Representativeness of the target species (Section 8.1) for this fish tissue sampling effort was established based on:

- the recommendation of USEPA's *Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Volume 1: Fish Sampling and Analysis, Third Edition* (USEPA 2000),
- the input from the study's lead fisheries biologist, and
- approval by the USEPA Project Manager.

The representativeness goal for the sample collection effort will be satisfied by using experienced field biologists to ensure that the samples collected are actually of the type (composite and species) specified for this study, and are from the locations targeted for this study. The Field Team Leader, who must be an experienced fisheries biologist, will assess conditions in the field and determine the most appropriate locations to collect fish relative to the WWTP at that site. The location where each individual fish is collected will be recorded on the Field Record Form, and a sampling site diagram will give an overview of the entire site.

### *Completeness*

Completeness is defined as the percentage of measurements made that are judged to be valid according to specific criteria and entered into the data management system. To optimize completeness, every effort is made to avoid sample and/or data loss. Accidents during sample transport or lab activities that cause the loss of the original samples will result in irreparable loss of data, which will reduce the ability to perform analyses, integrate results, and prepare reports. Samples will be stored and transported in unbreakable (plastic) containers (i.e., insulated ice chests). All sample processing (i.e., compositing, filleting, homogenization) will occur in a controlled environment within the laboratory, not in the field. The assignment of a set of specific sample numbers (Section 6.0) that have undergone chain-of-custody inspection makes it less likely for the sample preparation laboratory to overlook samples when preparing them for processing.

Percent completeness (%C) for measurement parameters can be defined as follows:

$$\%C = \frac{v}{T} \times 100$$

Where  $v$  = the number of measurements judged valid and  
 $T$  = the total number of measurements.

Completeness, in the case of this project, is the number of valid samples collected relative to the number of samples that are planned to be collected. Since this is a pilot study with a limited number of samples, the completeness goal for this project is 100%. It should be noted that sample locations and numbers may change over the course of the study, based on local conditions (e.g., accessibility of target sites) and the availability of target fishes (e.g., natural biological abundance or distribution). Any and all changes must be approved by the USEPA Project Manager, and approved changes must be considered when assessing completeness. The completeness goal is achieved when five effluent-dominated sites and one reference quality site found to contain target fishes are sampled, and the fish tissue samples are shipped with no errors in documentation or sample handling procedures. Completeness will be documented in the Field Sampling QA Report and Final Study Report.

### *Comparability*

Comparability is an expression of the confidence with which one data set can be compared with another. Comparability is dependent on the proper design of the sampling program and on adherence to accepted sampling techniques, standard operating procedures, and quality assurance guidelines. For the fish tissue collection task, comparability of data will be accomplished by standardizing the sampling season, the field sampling methods, and the field training as follows:

- All samples will be collected during the summer or fall of 2006 (August-November). Adjustments to this schedule may be necessary (based on availability of sampling personnel and equipment, and/or weather and water conditions); however, all adjustments must be approved by the USEPA Project Manager.
- All samples will be collected and prepared for shipment according to standard operating procedures contained in this QAPP (Appendix B). These procedures are consistent with the recommendations of USEPA's *Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Volume 1: Fish Sampling and Analysis, Third Edition* (USEPA 2000).
- All field personnel involved with sampling will have adequate training and appropriate experience (Section 5.0).

## **5.0 SPECIAL TRAINING REQUIREMENTS/CERTIFICATION**

Each Field Sampling Team is required to have the necessary knowledge and experience to perform all field activities. This includes both knowledge and experience in the collection and identification of fishes and in the use of fisheries sampling gear specified for the study. It also includes training in project-specific sample collection and handling procedures. The field sampling crews will be composed of contracted biologists with a strong technical background in fisheries sampling activities. Each Field Sampling Team will consist of one experienced fisheries biologist (that must have experience with the array of fisheries sampling gear types to be used) and field staff to assist with sample collection and processing. At some sites, the contracted biologists may enlist the aid of WWTP staff, state fisheries biologists, or other local personnel to provide logistical support and assist with sample collection. In these cases, each participant will attend an on-site training session led by the Tetra Tech Task Leader (an experienced fisheries biologist).

## **6.0 DOCUMENTATION AND RECORDS**

Thorough documentation of all field sample collection and handling activities is necessary for proper processing in the laboratory and, ultimately, for the interpretation of study results. Field sample collection and handling will be documented in writing (for each sampling site) using the following forms and labels:

- a Field Record Form that contains information about each individual specimen and sampling site (Appendix B),
- a Sample Identification Label that accompanies and identifies each sample (Appendix B),
- a Chain-of-Custody Label that seals each sample container, and
- a Chain-of-Custody Form that provides constant tracking information for all samples (Appendix B).

A detailed description of each sample collected by each Field Sampling Team will be recorded on a Field Record Form (Appendix B). The form will document the sampling date, time, sampler's name, sampling site location/description, and sample description (count, length, and weight of each specimen). The Field Record Form will also contain a unique tracking code (i.e., composite sample identification code) that will be used to identify each record. The nine-character code will include:

- state of collection (two-character abbreviation),
- year of collection (two-number abbreviation),
- site identification (three-digit city code from Appendix A),

- composite number (1 through 6)
- tissue fraction to be completed by laboratory (“L” for liver or “F” for fillet)

The Field Record Form will be produced as a three-page carbonless copy form, with one copy retained by the sampler, and the other two included in the sample shipment to the laboratory (i.e., one for the laboratory, and one for the Tetra Tech Task Leader). All entries will be made in ink and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark, which is initialed and dated by the sampler/recorder.

A Sample Identification Label will be completed (Appendix B) to accompany each sample throughout the chain of custody. The label will document the project name, sampling site location, sampling date and time, the sampler’s name, the nine-character tracking code, and the specimen number (e.g., 01 through 05). All entries will be made in indelible ink and will coincide with specimen and sample information on the Field Record Form. Descriptions and definitions of all field data elements required in the Field Record Form and Sample Identification Label are provided in Appendix D.

Proper chain-of-custody procedures are necessary for tracking sample possession from field to laboratory. Chain-of-Custody Forms (Appendix B) will accompany each shipment of samples and will document sample identity (coinciding with information on the field record), sampler relinquishment date and time, and laboratory receipt date and time. Chain-of-Custody Forms will be produced as four-page carbonless copies, with one copy for the sampler, and three for shipment to the laboratory (i.e., one for the laboratory, one for the Tetra Tech Task Leader, and one for distribution to the CSC data review team). Chain-of-Custody Labels will seal each sample container following packing operations in the field, and will include the signature of the sampler and the date and time sealed. All Chain-of-Custody Label and Form entries will be made in ink. Field sampling teams must notify the laboratory by telephone of an incoming shipment.

Samples will be shipped from the field to the laboratory via priority, overnight express delivery service. Copies of all shipping airbills will be retained by the laboratory. Specification for retention of field samples by the receiving location is outside the scope of this document. While in storage, it is recommended that samples held for analysis be stored with the original labeling materials.

Sampling activities will conclude with the development of a field collection effort summary (i.e., detailed listing of all sampling participants, sampling locations, and specimens collected) and Field Sampling QA Report by Tetra Tech and review of the summary by the USEPA Project Manager. Following USEPA Project Manager approval, the summary will be used to document and report back to study participants the collective sampling progress. Tetra Tech will maintain a file as a repository for information used in the preparation of the field collection summary throughout the duration of the study. The following information will be included:

- any documents prepared for the study,

- contract and work assignment information,
- project QAPP,
- results of technical reviews, data quality assessments, field observations, and audits,
- communications (memoranda; internal notes; telephone conversation records; letters; meeting minutes; and all written correspondence between Tetra Tech, USEPA, and other project team personnel, subcontractors, suppliers, or others),
- maps, photographs, and drawings, and
- studies, reports, and documents pertaining to the project.

If any change(s) in this QAPP is(are) required during the study, a memo will be sent to each person on the distribution list describing the change(s), following approval by the USEPA Project Manager. Any and all memos announcing changes must be attached to the QAPP.

All documents and records prepared for this project will be maintained by USEPA and Tetra Tech during the project, and retained for a period of three years following completion of the project (unless otherwise directed by USEPA).

## **B. DATA ACQUISITION**

### **7.0 SAMPLING PROCESS DESIGN**

The objective of the PPCP Fish Tissue Pilot Project is to investigate the occurrence of a broad suite of PPCPs (39 compounds) in the tissue of harvestable sized adult freshwater fish that are typically consumed by wildlife and humans.

In so doing, the study will provide the following types of information:

- the potential for the target PPCP compounds to bioaccumulate in fish muscle and liver tissue, and
- data to answer questions concerning the occurrence of these compounds in fish and the potential for human exposure through fish consumption.

For the purposes of this study design, the target population will be effluent-dominated streams associated with WWTPs within the contiguous United States. The streams in this study must have a viable fish population of a resident species which will spend most of its life stages within the effluent-dominated waters. A total of five locations will be sampled, plus one reference site.

## 7.1 Sample Type

To meet the study objectives, the PPCP Fish Tissue Pilot Project will include composite sampling of fish fillets and fish livers from each sample site. Six composite samples will be collected at each site. At least three adult individuals will be collected per composite, all of which will be large enough to provide sufficient tissue for analysis of the group of target analytes. It has been determined that at least 30 grams of edible fillet tissue and 10 grams of liver tissue will be required from the composites to allow for analysis of all target analytes. Based on the recommendations of USEPA's *Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Volume 1: Fish Sampling and Analysis, Third Edition* (USEPA 2000) and methods applied in USEPA's National Lake Fish Tissue Study (USEPA 2006), fish used in a composite sample must meet the following criteria:

- all be of the same species,
- satisfy any legal requirements of harvestable size or weight, or at least be of consumable size if no legal harvest requirements are in effect,
- be of similar size so that the smallest individual in a composite is no less than 75% of the total length of the largest individual,
- be collected at the same time (i.e., collected as close to the same time as possible but no more than 1 week apart) [**Note:** This assumes that a sampling crew was unable to collect all fish needed to prepare the composite sample on the same day. If organisms used in the same composite are collected on different days (no more than 1 week apart), individual fish will be frozen until all the fish to be included in the composite are available for delivery to the laboratory.], and
- be collected in sufficient numbers (at least 3 per composite) and of adequate size (at least 3 harvestable size adult specimens that collectively will provide greater than 30 grams of edible tissue and 10 grams of liver tissue) to allow analysis of recommended target analytes.

Individual organisms used in composite samples must be of the same species because of notable differences in the species-specific bioaccumulation potential. Accurate taxonomic identification is essential in preventing the mixing of closely related species with the target species. Under no circumstance should individuals from different species be used in a composite sample.

Fish for this project are being sampled from wastewater treatment plant effluent-dominated streams. Earlier reconnaissance may indicate that appropriate fish should be available at a site, but it is possible that inadequate numbers of target species meeting the sample criteria will be found when the site is fished. If this situation were to occur, the Field Sampling Leader will contact the USEPA Project Manager to discuss possible options, which include collecting a different size or species of fish, sampling a site farther downstream, or sampling an alternate location.

## **7.2 Sampling Period**

Field sampling will be conducted during the period when water and weather conditions are conducive to safe and efficient field sampling. For this study, the sampling period is from summer to early fall, since lipid content is usually highest and water levels are usually lowest at that time. Where possible, sampling should not occur during the spawning period of the particular target species being sought. With these recommendations in mind, and considering the geographic extent of the study area (i.e., range of latitudes and longitudes) the field sampling period will begin in August and last through November. Any adjustments to this schedule must be approved by the USEPA Project Manager.

## **7.3 Sample Frame**

For the purposes of this study, the target population will be effluent-dominated streams which serve as receiving waters for WWTPs within the contiguous United States. WWTPs using primary, secondary, and tertiary treatment methods and discharging to a stream or river are included in the sample frame. The streams in this study must also have a viable fish population of a resident species that is subject to effluent exposure for most of its life cycle.

## **7.4 Selection of Sampling Sites**

Sites were targeted (Appendix A) in mid- to large-sized cities representing diverse geographic regions of the country. Information on WWTP design capacity, average discharge, and in-stream waste concentration was collected for each candidate site through research of publicly accessible data (i.e., NPDES permits, WWTP websites, USGS flow data) and through phone calls to state officials and permitting agencies. Once this information was compiled, the list of candidate sites was used by the EPA project team to select a group of priority sites. The site selection criteria that were used are:

- High effluent flow versus ambient flow
- High population density
- Large fraction of elderly residents
- Large volume of PPCP sales/consumption (higher income brackets as surrogate)
- Fish availability

In addition, fisheries information was compiled for each candidate site. This was accomplished by reviewing published fisheries reports and obtaining first-hand information from state fisheries personnel. The site list was further narrowed down to 12 priority sites which could potentially support (via availability of resident species and tissue biomass) the intended sampling. The five top priority sites were selected from the 12 priority candidates to represent diverse geographic regions of the country.

## 8.0 SAMPLING METHODS

### 8.1 Target Species

Field sampling procedures will follow the recommendations of USEPA's *Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Volume I: Fish Sampling and Analysis, Third Edition* (USEPA 2000). According to the guidance, the primary criteria for selecting target fishes are that the species:

- are commonly consumed in the study area,
- may potentially accumulate high concentrations of chemicals, and
- have a wide geographic distribution.

Secondarily, the target species should be:

- easy to identify,
- abundant,
- easy to capture, and
- large enough to provide adequate tissue for analysis (i.e., harvestable size adult specimens that as a composite of at least three fish will provide at least 30 grams of edible tissue and 10 grams of liver tissue for analysis).

A single species of fish will be collected from each site. Suggested target species are listed in Table 3 in order of preference (adapted from USEPA 2000). Additional target species may be added to the list of preferred targets on an as-needed basis, following discussion with the USEPA Project Manager and/or the Tetra Tech Task Leader. The criteria listed above must be considered when selecting target species other than those listed in Table 3. Every effort will be made to collect the desired species and number (Section 8.2) of fish; however, the outcome of field sampling efforts will ultimately depend on the natural diversity and abundance of fish in the study streams.

### 8.2 Composite Sampling

The PPCP Fish Tissue Pilot Project will involve composite sampling of fish. Composite samples are cost-effective for estimating average tissue concentrations of target analytes in target species populations, and compositing ensures adequate sample mass for analysis of all target analytes. Six single-species composites will be collected from each target stream. Each composite will consist of at least three fish of adequate size (i.e., adult specimens that collectively will provide at least 30 grams of edible tissue and 10 grams of liver tissue) to allow analysis of the target analytes. Fish retained for a composite sample must meet the following criteria:

- all be of the same species,

- satisfy any legal requirements of harvestable size (or weight), or at least be of consumable size if no legal harvest requirements are in effect,
- be of similar size so that the smallest individual in a composite is no less than 75% of the total length of the largest individual, and
- be collected at the same time, i.e., collected as close to the same time as possible, but no more than one week apart (**Note:** Individual fish may have to be frozen until all fish to be included in the composite are available for delivery to the sample preparation laboratory).

Accurate taxonomic identification is essential in this study to define the organisms that have been composited and submitted for analysis. Under no circumstances should individuals from different species be used in a single composite sample. Ideally, the target species composite should focus on the larger individuals commonly harvested by the local population.

**Table 3.** Recommended Target Species for Inland Freshwaters (in Order of Preference).

	Family name	Common name	Scientific name
Predator/Gamefish Species (in order of preference)	<i>Centrarchidae</i>	Largemouth bass	<i>Micropterus salmoides</i>
		Smallmouth bass	<i>Micropterus dolomieu</i>
		Black crappie	<i>Pomoxis nigromaculatus</i>
		White crappie	<i>Pomoxis annularis</i>
	<i>Salmonidae</i>	Brown trout	<i>Salmo trutta</i>
		Rainbow trout	<i>Oncorhynchus mykiss</i>
		Brook trout	<i>Salvelinus fontinalis</i>
Bottom-dwelling Species (in order of preference)	<i>Cyprinidae</i>	Common carp	<i>Cyprinus carpio</i>
	<i>Ictaluridae</i>	Channel catfish	<i>Ictalurus punctatus</i>
		Blue catfish	<i>Ictalurus furcatus</i>
		Brown bullhead	<i>Ameiurus nebulosus</i>
		Yellow bullhead	<i>Ameiurus natalis</i>
	<i>Catostomidae</i>	White sucker	<i>Catostomus commersonii</i>

### 8.3 Sample Collection

The field objective is for sampling teams to obtain six representative composite samples from each stream selected for the PPCP Fish Tissue Pilot Project. To obtain a representative sample of the target species in each stream (and particularly in larger waterbodies), field teams will consider factors such as habitat and presence of contaminant gradients in planning sampling locations for the target stream. Prior to sampling, field teams will determine habitats suitable for target species, then sample those habitats in the stream reach located downstream from the WWTP outfall.

Fish collection methods can be divided into two major categories, active and passive. Each has advantages and disadvantages. Active collection methods employ a wide variety of sampling devices including electrofishing units, seines, trawls, and angling equipment (hook and line). Although active collection requires greater fishing effort, it is usually more efficient than passive collection for covering a large number of sites and catching the relatively small number of individuals needed from each site for tissue analysis. The active collection methods generally require more field personnel and more expensive equipment than passive collection methods. Passive collection methods employ a wide array of sampling devices, including gill nets, fyke nets, trammel nets, hoop nets, pound nets, and d-traps. Passive collection methods generally require less fishing effort than active methods, but normally yield a much greater catch than would be required for a contaminant monitoring program. They are also time consuming to deploy. Passive collection devices (e.g., gill nets) must be checked frequently (e.g., at least once every 24 hours) to ensure a limited time lag between fish entrapment and sample preparation/preservation.

Sampling Teams dedicated to the PPCP Fish Tissue Pilot Project will be equipped with an array of both active and passive gears to ensure the collection of the desired target numbers and species of fish. Selection of the most appropriate gear type(s) for a particular target stream will be at the discretion of the experienced on-site fisheries biologist. Contractor-affiliated Sampling Teams will be responsible for providing fisheries sampling gear, supplies, and sampling vessels. Sampling Teams must also have access to back-up gear or next-day replacement (through local suppliers or Fed-Ex) of malfunctioning gear. A list of equipment and expendable supplies is provided in Table 4. Sample collection, packaging, and shipment methods are presented as Standard Operating Procedures (Appendix B).

As soon as fish are obtained via active collection methods, or removed from passive collection devices, they must be identified to species. Species identification will be conducted only by experienced personnel knowledgeable of the taxonomy of species in the waterbodies included in the PPCP Fish Tissue Pilot Project. The person handling the fish will wear clean nitrile gloves. Nontarget species collected by the field team will be returned to the water. Individuals of the selected target species will be rinsed in distilled water to remove any foreign material from the external surface and placed in clean holding containers (livewell, buckets, etc.) to prevent contamination. Each fish of the selected target species will be measured to determine total body length (mm). Maximum body length is measured, i.e., the length from the anterior-most part of the fish to the tip of the longest caudal finray (when the lobes of the caudal fin are depressed dorsoventrally). Each fish of the selected target species will be weighed. When sufficient numbers of the target species have been identified to make up a suitable composite sample (i.e., at least 3 individuals meeting the size criteria presented in Section 8.2), the species name, specimen lengths, specimen weights, and all other site and sampling information will be recorded on the Field Record Form (Appendix B).

**Table 4.** Equipment and Supply Checklist for Fish Tissue Sampling.

Equipment and Supplies	Check
1. Sampling vessel (including boat, motor, trailer, oars, gas, and all required safety equipment) <sup>(a)</sup>	
2. Electrofishing equipment - OPTIONAL (including variable voltage pulsator unit, generator, electrodes, wiring cables, dip nets, protective gloves, protective boots, and all necessary safety equipment) <sup>(a)</sup>	
3. Nets - OPTIONAL (including trawls, seines, gill nets, fyke nets, trammel nets, hoop nets, pound nets, trap nets) <sup>(a)</sup> (Note: No tarred nets, and all nets need to be clean and free of potential contaminants)	
4. Angling equipment - OPTIONAL (including fishing rods, reels, line, terminal tackle, trot lines) <sup>(a)</sup>	
5. Coast Guard-approved personal floatation devices	
6. Maps of target rivers/streams and access routes	
7. Global Positioning System (GPS) unit	
8. Flow meter (including associated calibration supplies) <sup>(a)</sup>	
9. Livewell and/or buckets	
10. Measuring board (millimeter calibration)	
11. Scale (milligram calibration)	
12. Ice chests (Note: Each ice chest needs to be washed with detergent and rinsed with distilled water)	
13. Aluminum foil (solvent-rinsed and baked)	
14. Heavy-duty food grade polyethylene tubing	
15. Large plastic (composite) bags	
16. Knife or scissors	
17. Clean nitrile gloves	
18. Field Record Forms	
19. Sample Identification Labels	
20. Chain-of-Custody Forms	
21. Chain-of-Custody Labels	
22. Scientific collection permit	
23. Dry ice	
24. Heavy cloth or leather gloves for handling dry ice	
25. Black ballpoint pens and/or waterproof markers	
26. Clipboard	
27. Packing/strapping tape	
28. Overnight courier airbills	
29. Plastic cable ties	
30. First aid kit and emergency telephone numbers	
31. Distilled water	

<sup>(a)</sup> Selection and exact specifications at the discretion of the experienced on-site fisheries biologist.

## 9.0 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

### 9.1 Sample Handling

Clean nitrile gloves must be worn during the entire sample handling process, beginning with removing the fish from the sampling gear. Individuals of the selected target species must be rinsed in distilled water to remove any foreign material from the external surface. After initial

processing to determine species and size, each of the fish found to be suitable for the composite sample will be individually wrapped in extra heavy-duty aluminum foil (i.e., solvent-rinsed, oven-baked sheets). For specimens with sharp fins, spines may be broken (via gloved hands or with the use of a tool covered with the aluminum foil) to prevent perforation of the wrapping materials. The broken section of the fins must be included with the fish sample. A Sample Identification Label (Appendix B) will be prepared for each aluminum foil-wrapped specimen. Each foil-wrapped fish will be placed into a waterproof plastic tubing that will be cut to size to fit the specimen (i.e., heavy duty food grade polyethylene tubing) and each end of the tubing will be sealed with a plastic cable tie. The completed Sample Identification Label will be affixed to the cable tie, and the entire specimen package will be “double-bagged” (i.e., placed inside a large plastic bag with all the specimens of the same composite from that site and sealed with another cable tie). Once packaged, samples should be immediately placed on dry ice in an insulated ice chest or cooler for shipment. If samples will be carried back to a laboratory or other facility to be frozen before shipment, wet ice can be used to transport wrapped and bagged fish samples in the coolers to that laboratory or facility. If possible, all of the specimens in a composite sample should be kept together in the same shipping container (ice chest or cooler) for transport. Sampling Teams have the option, depending on site logistics, of:

- shipping the samples packed on dry ice (in sufficient quantities to keep samples frozen for up to 48 hours – at least 50 lbs. per cooler), via priority overnight delivery service (i.e., Federal Express), so that they arrive at the laboratory within less than 24 hours from the time of sample collection, or
- freezing the samples within 24 hours of collection (at  $\leq -20^{\circ}\text{C}$ ), and storing the frozen samples until shipment within 1 week of sample collection (frozen samples will subsequently be packed on dry ice in an insulated ice chest or cooler and shipped to the laboratory via priority overnight delivery service to arrive within less than 24 hours from time of shipment).

The time of sample collection, relinquishment by the sample team, and time of their arrival at the laboratory must be recorded on the Chain-of-Custody Form (Appendix B). Field Sampling Teams should avoid shipping samples for weekend delivery to the laboratory unless prior plans for such a delivery have been agreed upon with the laboratory.

## **9.2 Sample Integrity**

A critical requirement of the PPCP Fish Tissue Pilot Project is the maintenance of sample integrity from the time of collection to the shipment and arrival at the final destination. Sample integrity is maintained by preventing the loss of contaminants that might be present in the sample and by taking precautions to avoid possible introduction of contaminants during handling. The loss of contaminants can be prevented in the field by ensuring that the sample collected remains intact, i.e., sample collection procedures should be performed with the intention of minimizing the laceration of fish skin. Once a sample is collected, sample integrity is maintained through careful and controlled sample handling, storage, and preservation procedures (Section 9.1).

Preventable sources of extraneous contamination can include the sampling gear, oils and greases on boats, spilled fuel, skin contact, contact with soil or sand, boat motor exhaust, and other potential sources. All potential sources should be identified before the onset and during sample collection, and appropriate measures should be taken to minimize or eliminate them. Examples of preventative measures include the following:

- Collection nets should be free of any potential contaminants.
- The use of tarred collection nets is prohibited.
- Boats should be positioned so that engine exhaust does not fall on the deck area where samples are being handled.
- Ice chests and other sample storage containers should be scrubbed clean with detergent and rinsed with distilled water prior to use.
- Samples should not be placed directly on dry ice, but should be stored inside foil, plastic tubing (i.e., heavy-duty food grade polyethylene tubing as per Section 9.1), and plastic garbage bags first.
- Proper gloves (clean nitrile gloves) should be used when handling samples.

### **9.3 Custody Requirements**

As soon as possible following collection, the Sampling Team will begin the process of identifying, labeling, packaging, and storing the sample(s). Each sample will be identified and tracked with a unique numbering scheme as described in Section 6.0. This nine-character composite code followed by a two-digit specimen number will identify each sample on all documentation and records including the following:

- Field Record Form,
- Sample Identification Label, and
- Chain-of-Custody Form.

Each sample (i.e., individual fish) will be labeled by affixing a Sample Identification Label (Appendix B) as per the instructions in Section 9.1. All sample label entries will be made with black indelible ink. The sample label will accompany each sample throughout the chain-of-custody. Each sample label will include the following information:

- project name (USEPA PPCP Fish Tissue Pilot Project),
- site identification (receiving waterbody's name),
- specimen number (at minimum, 01 through 03),

- composite code (nine-digit code as in Section 6.0),
- date of sample (month/day/year),
- time of collection (military time/time zone/standard or daylight savings time),
- preservative used (dry ice or wet ice/frozen), and
- collector's name (field team leader).

Detailed documentation of the samples collected in the field (for shipment to the laboratory) and information about the collection location will be recorded on a Field Record Form (Appendix B). One form must be completed for each sample composite. One page of the three-page carbonless copy form (Section 6.0) will be retained by the sampler, and the other copies will be included with sample shipment to the laboratory. (The laboratory will retain one copy, and be responsible for forwarding one copy to the Tetra Tech Task Leader.) All entries will be made in waterproof black ink and no erasures will be made. Each form will have the proper entry requirements, which includes the following information:

- composite code (nine digits as per Section 6.0),
- sampling date (month/day/year),
- time of collection (military time),
- collection method (e.g., electrofisher),
- collector's name (printed and signed),
- collector's affiliation, address, and telephone number,
- site location (county name and site coordinates),
- facility name,
- site name (receiving waterbody's name),
- site description (location, area sampled),
- estimated average depth (meters [m]),
- estimated average width (meters [m]),
- stream flow (meters/second [m/sec]),

- average stream flow (meters/second [m/sec])
- fish species (common name),
- length (millimeters [mm]) of each specimen,
- weight (grams [g]) of each specimen,
- location, date and time of collection for each specimen, and
- a simple sketch of the sampling site and sample collection points.

All samples and composites will be transferred to the receiving laboratory under chain of custody. The Chain-of-Custody Form (Appendix B) acts as a record of sample shipment and a catalog of the contents of each shipment (coinciding with information on the field record). The forms will be produced as four-page carbonless copies. One copy of the completed form will be retained by the sampler and the remaining three will be inserted into a sealable plastic bag and included in the shipment of each cooler to the laboratory. The laboratory will sign the form on receipt of the samples, keep one copy and send the other two copies to the Tetra Tech Task Leader and one to the CSC data review team. All Chain-of-Custody Form entries will be made in waterproof black ink and will include:

- the USEPA Project Manager's name, address and telephone number (refer to the Distribution List, pg. vi),
- sampler's name and telephone number,
- project name (USEPA PPCP Fish Tissue Pilot Project),
- page number (e.g., 1 of 1),
- sample location (receiving waterbody's name),
- collection date and time,
- composite code (nine-digit) and specimen number (two-digit),
- preservative (dry ice or wet ice/frozen),
- number of containers,
- type of analysis required (39 PPCP target analytes),
- sampler's signature, sample date, and time,
- sampler relinquishment date and time,

- laboratory recipient signature, and
- laboratory receipt date and time.

Immediately following the packing of each shipping container (Section 9.1), each container (ice chest) will be secured with packaging tape and sealed with a Chain-of-Custody Label. The Chain-of-Custody Label must contain the signature of the sampler and the date and time written in ink. The seal must be affixed such that the shipping container cannot be opened without breaking the seal (e.g., label adhered across the ice chest latch), so as to protect and document the integrity of the contents from field to laboratory.

## **10.0 ANALYTICAL METHODS REQUIREMENTS**

Samples will be shipped (Section 9.1) under chain of custody to locations designated by the USEPA Project Manager for processing and analytical testing. Sample processing and analytical testing and methods are outside the scope of this QAPP and therefore are not addressed herein, but will be discussed in the Analytical Activities QAPP.

## **11.0 QUALITY CONTROL REQUIREMENTS**

Data quality is addressed, in part, by consistent performance of valid procedures documented in the standard operating procedures (Appendix B). It is enhanced by the training and experience of project staff (Section 5.0) and documentation of project activities (Section 6.0). This QAPP will be distributed to all sampling personnel. Field Team Leaders will be required to read the QAPP, and verify that they read or viewed the materials and understood the procedures and requirements.

Data quality will be addressed in the field through a thorough check of the Field Record Forms by the Field Team Leader. The Field Team Leader will review each form for completeness and accuracy of the recorded data. It is important to complete this step while still in the field so that any unclear data can be verified and corrected. The Field Record Forms will be reviewed again by Tetra Tech upon receipt, and a Data Review Form will be completed for each Field Record Form. The Data Review Form documents the completeness and accuracy of all of the recorded data; if corrections or additional data are necessary, the revisions will be documented on the Data Review Form.

## **12.0 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS**

All field equipment will be inspected prior to sampling activities to ensure that proper use requirements are met (e.g., boats or electrofishers are operating correctly, nets are without defects, flow meter properly calibrated). Inspection of field equipment will occur well in advance of the field operation to allow time for replacement or repair of defective equipment,

and the field team will be equipped with proper backup equipment to prevent lost time on site. One member of each field team should gather and inspect all equipment on the equipment and supply checklist (Table 4) prior to each sampling event.

### **13.0 INSTRUMENT CALIBRATION AND FREQUENCY**

All flow meters used by field teams will be calibrated according to the manufacturer's operating instructions, on a daily basis, while in use (Appendix C).

### **14.0 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES**

Careful and thorough planning is necessary to ensure the efficient and effective completion of the field sample collection task. A general checklist of field equipment and supplies is provided in Table 4. Sampling gear and supplies will be provided by each field team. It will be the responsibility of each field team to gather and inspect the necessary sampling gear and sample packaging and shipping supplies prior to the sampling event.

### **15.0 DATA ACQUISITION REQUIREMENTS (NONDIRECT MEASUREMENTS)**

Nondirect measurements will include identification and/or verification of each sample site location. Names of the target streams and addresses of the associated WWTPs will be provided to the field sampling teams. Appropriate maps (USGS quadrangle, gazetteer, street atlas) will be used to locate the site and verify the receiving waters that are being sampled.

### **16.0 DATA MANAGEMENT**

Samples will be documented and tracked via Sample Identification Labels, Field Record Forms, and Chain-of-Custody Forms (Section 6.0). Field team leaders will be responsible for reviewing all completed field forms. Any corrections should be noted, initialed, and dated by the reviewer (Section 6.0). As mentioned in Section 6.0, Field Record Forms and Chain-of-Custody Forms will each be prepared and replicated in the field, via multiple page "carbonless copy" forms. The sampler will retain one copy each of the Field Record and Chain-of-Custody Forms, and the remaining copies will be delivered to the laboratory with the samples. Shipment of samples to the laboratory (Section 9.1) must be conducted by a delivery service that provides constant tracking of shipments (e.g., Federal Express). Laboratory sample log-in and data management procedures are beyond the scope of this QAPP.

The laboratory will retain one copy of each Field Record Form and Chain-of-Custody Form, and will forward the original form to the Tetra Tech Task Leader. Tetra Tech will perform a data quality control check on the Field Record Form and forward the original form and the Data Quality Control form to the USEPA Project Manager. Tetra Tech will retain a copy of the Field

Record Form and Data Quality Control form. All form copies obtained by Tetra Tech will be maintained in a project file during the active phase of the project, and for a period of 3 years following completion of the project (unless otherwise directed by USEPA). Upon completion of sampling activities, Tetra Tech will develop a field collection effort summary (i.e., a detailed listing of all sampling participants, sampling locations, and specimens collected) based on information recorded by all Sampling Teams on the Field Record Forms. The Field Record Form data will be entered into an Excel® spreadsheet to create the summary. All data entries will be checked for errors in transcription and computer input by a minimum of two persons. If there is any indication that requirements for sample integrity or data quality have not been met, the Tetra Tech QA Officer will be notified immediately (with an accompanying explanation of the problems encountered). All computer files associated with the project will be stored in a project subdirectory by Tetra Tech, and will be copied to disk for archive for the three years subsequent to project completion (unless otherwise directed by the USEPA Project Manager).

## **C. ASSESSMENT/OVERSIGHT**

### **17.0 ASSESSMENT AND RESPONSE ACTIONS**

Assessment activities and corrective response actions have been identified to ensure that sample collection activities are conducted as prescribed and that the measurement quality objectives and data quality objectives established by USEPA are met. The QA program under which this project will operate includes performance and system audits with independent checks of the data obtained from sampling activities. Either type of audit could indicate the need for corrective action. The essential steps in the program are as follows:

- identify and define the problem,
- assign responsibility for investigating the problem,
- investigate and determine the cause of the problem,
- assign and accept responsibility for implementing appropriate corrective action,
- establish effectiveness of and implement the corrective action, and
- verify that the corrective action has eliminated the problem.

Immediate corrective actions form part of normal operating procedures and are noted on project Field Record Forms. The most common corrective action of this type is the resolution of non-routine composite samples. Composite samples should contain at least three fish, and the smallest fish should be at least 75% of the length of the largest fish. If either of these conditions is not met, the sample is considered to be non-routine. Non-routine samples are not automatically eliminated from the study. Non-routine sample details must be documented in a memorandum to the USEPA Project Manager so that the sample can be evaluated to determine if

it is programmatically viable (e.g., sufficient tissue for analysis). The non-routine memo is submitted from the Tetra Tech Task Leader to the USEPA Project Manager. It lists each non-routine composite sample and describes species, number of specimens, and specimen lengths and weights. The sample is then evaluated by the USEPA Project Manager and the USEPA Project Statistician, who collectively decide whether it is suitable for inclusion and analysis. They may decide to do the following: accept non-routine composites that have less than three specimens, but provide sufficient tissue for analysis; or reject individual specimens or entire composites that are outside of program parameters. After the USEPA Project Manager and the USEPA Project Statistician evaluate and reach a decision regarding the non-routine composites, sample details and resolution elements are recorded on a Resolved Action Form (Appendix E).

Some problems that arise during the study may require more formalized, long-term corrective action. In the event that quality problems requiring attention are identified, the Tetra Tech Task Leader and/or Tetra Tech QA Officer will determine whether attainment of acceptable data quality requires either short- or long-term actions. Failure in an analytical system (e.g., performance requirements are not met) and corrective actions for those failures are beyond the scope of this QAPP.

Communication and oversight will proceed from Field Sampling Team Leaders (e.g., senior fisheries biologist) to the Tetra Tech Task Leader. The Tetra Tech Task Leader will oversee the review of all Field Record Forms upon receipt (using field sampling activities data review forms as presented in Appendix F), and will communicate the status of the sampling activities to the USEPA Project Manager on a weekly basis (at a minimum). The Tetra Tech Task Leader will immediately consult with the Tetra Tech QA Officer and USEPA Project Manager regarding any difficulties encountered during sample collection activities. The Tetra Tech QA Officer will initiate the corrective action system described above, documenting the nature of the problem in a system audit report and ensuring that the recommended corrective action is carried out.

The USEPA Project Manager and/or the Tetra Tech QA Officer will work with the Tetra Tech Task Leader to determine the best way to rectify the problem and obtain accurate and useable data. When corrective actions have been taken and a sufficient time period has elapsed that allows a response, the response will be compared with project goals by the USEPA Project Manager. The Tetra Tech QA Officer will verify that the corrective action has been appropriately addressed to eliminate the problem. The USEPA Project Manager in coordination with the USEPA Project Officer has the authority to stop work on the project if problems affecting data quality are identified that will require extensive effort to resolve. The USEPA Project Manager will consult with the USEPA QA Manager regarding any and all corrective actions and stop work orders.

Performance audits are qualitative checks on different segments of project activities, and are most appropriate for sampling, analysis, and data processing activities. Two field audits will be conducted in accordance with Agency requirements and availability of resources. Audits will be conducted at the Dallas, TX site and the West Chester, PA site (Appendix A). The USEPA QA Officer or authorized designee will conduct this audit. The field audit is designed to ensure that field activities are adequately observed and monitored for adherence to field sampling protocols. This gives the Project Management Team the opportunity to identify non-compliance issues and

to complete corrective actions. During the audit, the field observer will follow a standard field observation protocol as outlined in the Field Observation Plan (Appendix G), and complete a Field Observation Checklist (Appendix H). The auditor will complete a field procedural review checklist and immediately inform the sampling team members of any deviations from the procedures or other concerns.

Performance audit techniques include checks on sampling equipment, measurements, and the analysis of data quality using QC and spiked samples. Analytical performance audits are beyond the scope of this QAPP. The USEPA Project Manager and/or the Tetra Tech Task Leader will be responsible for overseeing work as it is performed, and periodically conducting QC checks during the sample collection phase of this project.

System audits are qualitative reviews of project activities to check that the overall quality program is functioning and that the appropriate QC measures identified in the QAPP are being implemented. The Tetra Tech QA Officer will conduct one internal system audit during the project and report the results to the USEPA Project Manager on Tetra Tech's standard Audit Report Form within thirty days. If QA/QC deficiencies are discovered, additional internal system audits will be conducted until the Tetra Tech QA Officer and the USEPA Project Manager conclude that overall project quality requirements are being met.

## **18.0 REPORTS TO MANAGEMENT**

Following completion of the system audits, the Tetra Tech QA Officer will prepare an Audit Report Form and submit copies to the USEPA Project Manager, the USEPA QA Coordinator, and the USEPA QA Officer within thirty days of the audit.

Upon completion of weekly sampling activities, the Tetra Tech Task Leader will contact the USEPA Project Manager to summarize Field Sampling Team progress for the preceding week and submit a weekly progress report detailing the sampling activities and noting any concerns about sample quality and their resolution. Following completion of field sampling activities, Tetra Tech will prepare a field collection effort summary (i.e., detailed listing of all sampling participants, sampling locations, and specimens collected) and Field Sampling QA Report for review by the USEPA Project Manager. Following incorporation of USEPA Project Manager comments and final approval, the summary and Report will be used to report back to study participants to document the sampling effort for the study.

## **D. DATA VALIDATION AND USABILITY**

### **19.0 DATA REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS**

Data validation and review services provide a method for determining the usability and limitations of data, and provide a standardized data quality assessment. All Field Record Forms and Chain-of-Custody records will be reviewed by the Tetra Tech Task Leader for completeness

and correctness. Tetra Tech will be responsible for reviewing data entries and transmittals for completeness and adherence to QA requirements. Data quality will be assessed by comparing entered data to original data or by comparing results with the measurement performance criteria summarized in Section 4.2 to determine whether to accept, reject, or qualify the data. Results of the review and validation processes will be reported to the USEPA Project Manager.

## **20.0 VALIDATION AND VERIFICATION METHODS**

All Field Record Forms and Chain-of-Custody records will be reviewed by the Tetra Tech Task Leader upon receipt. The Tetra Tech QA Officer or authorized designee will review the Field Record Forms and Chain-of-Custody records during the system audit. Any discrepancies in the records will be reconciled with the appropriate associated field personnel and will be reported to the USEPA Project Manager.

Analytical validation and verification methods are outside of the scope of this QAPP. The submission of samples to the laboratory will include Field Record Forms and Chain-of-Custody Forms documenting sampling time and date. This information will be checked by the receiving laboratory to ensure that holding times (Section 9.1) have not been exceeded. Violations of holding times will be reported (by the laboratory) to the USEPA Project Manager and the Tetra Tech Task Leader, and the USEPA Project Manager will discuss with the laboratory whether or not to issue a stop work order for analysis of that particular sample.

## **21.0 RECONCILIATION WITH DATA QUALITY OBJECTIVES**

As soon as possible following completion of the sample collection task, precision, accuracy, and completeness measures will be assessed by Tetra Tech and compared with the criteria discussed in Section 4.0. This will represent the final determination of whether the data collected are of the correct type, quantity, and quality to support their intended use for this project. Any problems encountered in meeting the performance criteria (or uncertainties and limitations in the use of the data) will be discussed with the USEPA Project Manager, and will be reconciled, if possible.

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## LITERATURE CITED

U.S. Environmental Protection Agency (USEPA). 2000. Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Volume 1: Fish Sampling and Analysis, Third Edition. U.S. Environmental Protection Agency, Office of Water, Washington, D.C. EPA-823-B-00-007.

U.S. Environmental Protection Agency (USEPA). 2001. EPA Requirements for Quality Assurance Project Plans (EPA/QA/R-5). U.S. Environmental Protection Agency, Office of Environmental Information, Washington, D.C. EPA/240/B-01/003.

U.S. Environmental Protection Agency (USEPA). 2006 (Revision). Quality Assurance Project Plan for Sample Collection Activities for a National Study of Chemical Residues in Lake Fish Tissue. U.S. Environmental Protection Agency, Office of Water, Office of Science and Technology, Washington, D.C.



## **Appendix A**

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### **Targeted Wastewater Treatment Plant Sites**

**Table A-1.** A list of candidate sampling sites for the PPCP Fish Tissue Pilot Project.

St.	City	Facility Name	Treatment	Receiving Water Name	County Name	Pop	PCS Design Capacity	Existing Flow MGD	% Effluent	% 65& older	Median Income
AZ	Phoenix (PHX)	91st Avenue WWTP	secondary	Salt River	Maricopa	1,418,041	165	153	100% <sup>1</sup>	8.1	\$41,207
IL	Chicago (CHI)	Northside WRD	Advanced secondary	North Shore Channel	Cook	5,376,741	333	234	100% <sup>3</sup>	10.3	\$38,625
TX	Dallas (DAL)	Dallas WWTP	tertiary	Trinity River†	Dallas	3,500,000	150		100% <sup>3</sup>	8.1	\$43,324
FL	Orlando (ORL)	Orlando-Iron Br Fac	Advanced Treatment I	Little Econlockhatchee	Seminole	442,542	40.00	36	64% <sup>2</sup>	11.3	\$35,732
PA	West Chester (WEC)	Taylor Run WWTP	secondary	Taylor Run	Chester	17,701	1.5		36 - 86%*	9.0	\$37,803
NM	Santa Fe (SAF)	Santa Fe WWTP	secondary	Santa Fe River	Santa Fe	68,041	9	8.5	100%	13.9	\$49,705
NV	Las Vegas (LAV)	City of Las Vegas WPCF	primary & secondary, removes ammonia	Las Vegas Wash‡	Clark	575,000	110	84	100% <sup>3</sup>	11.6	\$44,046
TX	San Antonio (SAN)	Dos Rios WRC	Advanced treatment & disinfection	Medina & San Antonio Rivers	Bexar	1,236,249	125	56.5	100%	10.4	\$36,214
VA	Lorton (LOR)	Noman M. Cole, Jr. Pollution Control Plant	tertiary	Pohick Creek	Fairfax	17,786	54	>40	no gauge data	7.9	\$81,050
TX	Denton (DNT)	Pecan Creek Water Reclamation Plant	secondary	Pecan Creek	Denton	98,288	21		100% <sup>4</sup>	7.9	\$35,422
NC	Raleigh (RAL)	Neuse River WWTP	tertiary	Neuse River	Wake	326,653	60	36.67	48 - 78%	8.3	\$46,612
DC	Washington DC (WDC)	Blue Plains STP	tertiary	Potomac River	DC	553,523	370	335	no gauge data ?	12.2	\$40,127

 = Priority  
 = Secondary

- \* Instream waste concentration from Tetra Tech's WERF field sampling data
- ◇ Gauge is upstream of the Potomac's confluence with the Anacostia River; Blue Plains STP is downstream of this confluence, so Potomac's discharge will be much greater than the gauge shows.
- 1 Information from Debra Daniel, AZ DEQ
- 2 Calculation based on data provided by Alex Trounov, Tt Fairfax
- 3 Flow is primarily made up of effluent discharged from multiple facilities ([http://ndep.nv.gov/docs\\_04/bwpc\\_nv0000060\\_fs.pdf](http://ndep.nv.gov/docs_04/bwpc_nv0000060_fs.pdf) & [www.epa.gov/osp/regions/emerpoll/howe.ppt](http://www.epa.gov/osp/regions/emerpoll/howe.ppt)).
- 4 During non-storm conditions, flows are comprised almost entirely of effluent; during summer months, water in Pecan Cr. is exclusively effluent (Brooks et. al., 2005).
- ‡ Natural drainage in the Las Vegas Valley and the receiving stream for all area surface water dischargers. The water in the wash is primarily treated wastewater from the Clark County Water Reclamation District, City of Las Vegas, & City of Henderson.
- † Recommendation from Scott Dyer, PGI

## **Appendix B**

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### **Standard Operating Procedure: Fish Tissue Sample Collection Procedures for a PPCP Fish Tissue Pilot Project**

## Standard Operating Procedure

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**Fish Tissue Sample Collection Procedures for a  
PPCP Fish Tissue Pilot Project**

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**Scope and Applicability:** This Standard Operating Procedure (SOP) must be followed by all Field Sample Collection Teams involved with the USEPA Office of Water's PPCP Fish Tissue Pilot Project. Adherence to the SOP will ensure that field sampling activities will be performed the same way every time, i.e., are standardized, for all sampling participants.

Fish tissue sample collection procedures are presented below as sequential steps, and include specific equipment, materials, and methods required to perform field sampling activities only.

**Responsibility and Personnel Qualifications:** This procedure may be used by any Field Sampling Teams that have been authorized by the USEPA Project Manager to collect fish for the PPCP Fish Tissue Pilot Project.

**References:** U.S. Environmental Protection Agency (USEPA). 1999. National Study of Chemical Residues in Lake Fish Tissue: Study Design. U.S. Environmental Protection Agency, Office of Water, Office of Science and Technology, Washington, D.C.

U.S. Environmental Protection Agency (USEPA). 2000a. Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories. Volume 1: Fish Sampling and Analysis. Third Edition. U.S. Environmental Protection Agency, Office of Water, Washington, D.C. EPA 823-B-00-007.

U.S. Environmental Protection Agency (USEPA). 2000b. Quality Assurance Project Plan for Sample Collection Activities for a National Study of Chemical Residues in Lake Fish Tissue. U.S. Environmental Protection Agency, Office of Water, Office of Science and Technology, Washington, D.C. EPA-823-R-02-005.

U.S. Environmental Protection Agency (USEPA). 2001. EPA Requirements for Quality Assurance Project Plans (EPA/QA/R-5). U.S. Environmental Protection Agency, Office of Environmental Information, Washington, D.C. EPA/240/B-01/003.

U.S. Environmental Protection Agency (USEPA). 2002. (revision). Field Sampling Plan for the National Study of Chemical Residues in Lake Fish Tissue. U.S. Environmental Protection Agency, Office of Water, Office of Science and Technology, Washington, D.C. EPA-823-R-02-004.

**Precautions:** Follow usual safety precautions for working in the field. Boats and/or electrofishing equipment should only be operated by qualified, experienced operators trained in their proper use. Each vessel must be equipped with the appropriate Coast Guard-required safety equipment (including personal floatation devices for each field team member). If electrofishing equipment is used for sample collection, each team member must be insulated from the water, boat, and electrodes via rubber boots and gloves.

**Equipment/Materials Checklist:**

Equipment and Supplies	Check
1. Sampling vessel (including boat, motor, trailer, oars, gas, and all required safety equipment) <sup>(a)</sup>	
2. Electrofishing equipment - OPTIONAL (including variable voltage pulsator unit, generator, electrodes, wiring cables, dip nets, protective gloves, protective boots, and all necessary safety equipment) <sup>(a)</sup>	
3. Nets - OPTIONAL (including trawls, seines, gill nets, fyke nets, trammel nets, hoop nets, pound nets, trap nets) <sup>(a)</sup> (Note: No tarred nets, and all nets need to be clean and free of potential contaminants)	
4. Angling equipment - OPTIONAL (including fishing rods, reels, line, terminal tackle, trot lines) <sup>(a)</sup>	
5. Coast Guard-approved personal floatation devices	
6. Maps of target rivers/streams and access routes	
7. Global Positioning System (GPS) unit	
8. Flow meter (including associated calibration supplies) <sup>(a)</sup>	
9. Livewell and/or buckets	
10. Measuring board (millimeter calibration)	
11. Scale (milligram calibration)	
12. Ice chests (Note: Each ice chest needs to be washed with detergent and rinsed with distilled water)	
13. Aluminum foil (solvent-rinsed and baked)	
14. Heavy-duty food grade polyethylene tubing	
15. Large plastic (composite) bags	
16. Knife or scissors	
17. Clean nitrile gloves	
18. Field Record Forms	
19. Sample Identification Labels	
20. Chain-of-Custody Forms	
21. Chain-of-Custody Labels	
22. Scientific collection permit	
23. Dry ice	
24. Heavy cloth or leather gloves for handling dry ice	
25. Black ballpoint pens and/or waterproof markers	
26. Clipboard	
27. Packing/strapping tape	
28. Overnight courier airbills	
29. Plastic cable ties	
30. First aid kit and emergency telephone numbers	
31. Distilled water	

<sup>(a)</sup> Selection and exact specifications at the discretion of the experienced on-site fisheries biologist.

**Procedures:**

1. Identify the target stream to be sampled using the USEPA Office of Water's Target Site List (Appendix A). Locate the target stream via the Wastewater Treatment Plant (WWTP) address provided in the Target Site List and USGS topographic maps (or equivalent maps).

2. Based on site reconnaissance, determine whether the target site meets the requirements of a suitable site for sampling for the purposes of this study, i.e., each site must:
  - be an effluent-dominated stream associated with a WWTP, and
  - have a viable fish population of a resident species that is subject to effluent exposure for most of its life cycle.

If the target site meets the above criteria, and if in the case of private property the landowner allows access/permission to sample the site, proceed with Step 3. If the site does not meet the requirements and/or if a private landowner denies access, record the problem and contact the USEPA Project Manager.

3. Assemble an array of both active and passive gear types, to ensure the collection of the desired target numbers and species of fish. Selection of the most appropriate gear type(s) for a particular target site will be at the discretion of the experienced on-site fisheries biologist. Detailed procedures for use or deployment of all possible gear types are not included here. However, if passive collection devices (e.g., gill nets) are used, they must be checked frequently (e.g., several times daily if possible, but at least every 24 hours) to ensure a limited time lag between fish entrapment and sample preparation. Sampling Teams must be qualified, experienced, and/or trained on the safe and effective use of each gear type selected.

4. Sampling gear will be selected and deployed to obtain samples of the targeted species. Suggested target species, listed in order of preference, are as follows:

	Family name	Common name	Scientific name
<b>Predator/Gamefish Species (in order of preference)</b>	<i>Centrarchidae</i>	Largemouth bass	<i>Micropterus salmoides</i>
		Smallmouth bass	<i>Micropterus dolomieu</i>
		Black crappie	<i>Pomoxis nigromaculatus</i>
		White crappie	<i>Pomoxis annularis</i>
	<i>Salmonidae</i>	Brown trout	<i>Salmo trutta</i>
		Rainbow trout	<i>Oncorhynchus mykiss</i>
		Brook trout	<i>Salvelinus fontinalis</i>
<b>Bottom-dwelling Species (in order of preference)</b>	<i>Cyprinidae</i>	Common carp	<i>Cyprinus carpio</i>
	<i>Ictaluridae</i>	Channel catfish	<i>Ictalurus punctatus</i>
		Blue catfish	<i>Ictalurus furcatus</i>
		Brown bullhead	<i>Ameiurus nebulosus</i>
		Yellow bullhead	<i>Ameiurus natalis</i>
	<i>Catostomidae</i>	White sucker	<i>Catostomus commersonii</i>

5. As soon as fish have been obtained via active collection methods (or removed from passive collection devices) they must be identified to species. Clean nitrile gloves must be worn during the sample handling process. Potential target species/individuals will be rinsed in distilled water to remove any foreign material from the external surface and placed in clean holding containers (e.g., livewells, buckets). Nontarget fishes or small specimens are returned to the stream.
6. Six composite samples of one target species will be retained from each target site. Each composite must consist of 3-4 fish of adequate size (i.e., adult specimens that collectively will provide greater than 30 grams of edible tissue and 10 grams of liver tissue) for analysis. Select fish for each composite based on the following criteria:
- all are of the same species,
  - all satisfy legal requirements of harvestable size (or weight), or at least be of consumable size if no legal harvest requirements are in effect,
  - all are of similar size, so that the smallest individual in a composite is no less than 75% of the total length of the largest individual, and
  - all are collected at the same time, i.e., collected as close to the same time as possible, but no more than one week apart (Note: Individual fish may have to be frozen until all fish to be included in the composite are available for delivery to the sample preparation laboratory).

Accurate taxonomic identification is essential in assuring and defining the organisms that have been composited and submitted for analysis. Under no circumstances should individuals from different species be used in a single composite sample.

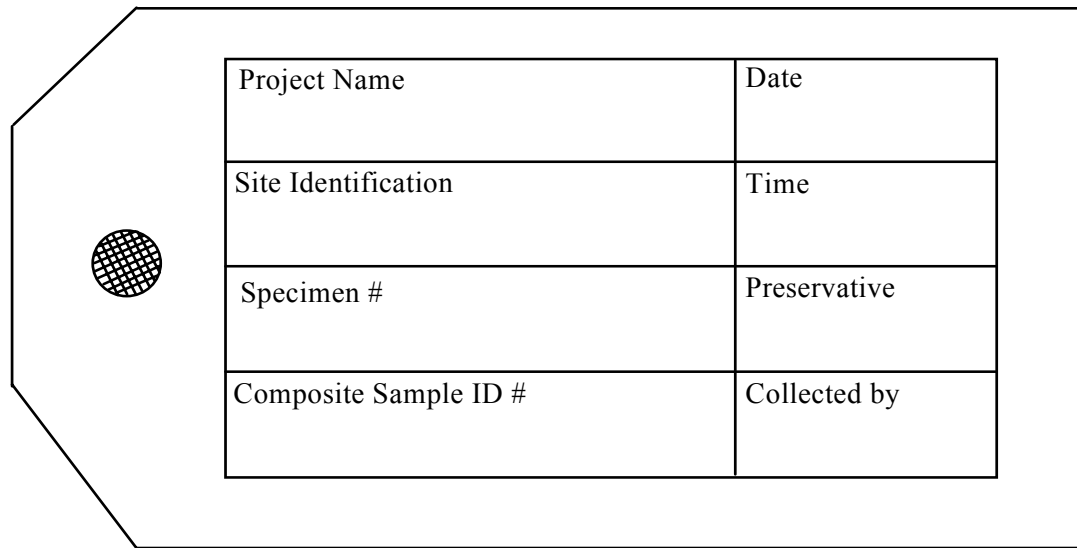
7. Following selection of 3-4 fish for each of the six composites that meet the above-listed criteria for compositing, measure each to determine total body length. Measure total length of each specimen in millimeters, from the anterior-most part of the fish to the tip of the longest caudal finray (when the lobes of the caudal fin are depressed dorsoventrally).
8. Measure total biomass of each specimen to the nearest gram, using an electronic scale or spring balance.
9. Record species retained, specimen length, biomass, location collected and sampling date and time on the Field Record Form (Figure 1) in black ink. Complete site location description portions of the form, and draw a simple sketch of the sampling area in the space provided. One Field Record Form will be completed for each composite collected from the target site.
10. Assign the unique nine-character composite sample ID number to each composite as directed on the Field Record Form (Figure 1):
  - state of collection (two-character abbreviation),
  - year of collection (two-number abbreviation),
  - site identification number (three-digit city code from Appendix A),
  - composite number (1 through 6)
  - tissue type (one character: F = fillet; L = liver)
11. Sign and date the Field Record Form.
12. Remove each fish retained for analysis from the clean holding container(s) (e.g., livewell) using clean nitrile gloves. Dispatch each fish using a clean wooden bat (or equivalent wooden device).
13. Wrap each fish in extra heavy-duty aluminum foil (solvent-rinsed, oven-baked sheets).
14. Prepare a Sample Identification Label (Figure 2) (in black ink) for each sample, ensuring that the label information matches the information recorded on the Field Record Form.
15. Cut a length of food grade tubing that is long enough to contain each individual fish and to allow extra length on each end to secure with cable ties. Place each foil-wrapped specimen into the appropriate length of tubing. Seal each end of the tubing with a plastic cable tie, and attach the appropriate Sample Identification Label.
16. Double-bag each entire specimen package, that is, place inside a large plastic bag with all specimens of the same composite from that site and seal with another cable tie.

17. As soon as each sample is packaged, place it immediately on dry ice for shipment. If samples will be carried back to a laboratory or other facility to be frozen before shipment, wet ice can be used to transport wrapped and bagged fish samples in the coolers to a laboratory or other interim facility.
18. If possible, keep all specimens designated for a particular composite in the same shipping container (ice chest) for transport.
20. Samples may be stored on dry ice for a maximum of 24 hours. Sampling teams have the option, depending on site logistics, of:
  - shipping the samples packed on dry ice in sufficient quantities to keep samples frozen for up to 48 hours, via priority overnight delivery service (e.g., Federal Express), so that they arrive at the laboratory within less than 24 hours from the time of sample collection, or
  - freezing the samples within 24 hours of collection at  $\leq -20^{\circ}\text{C}$ , and storing the frozen samples until shipment within 1 week of sample collection (frozen samples will subsequently be packed on dry ice and shipped to the laboratory via priority overnight delivery service).
21. Complete a Chain-of-Custody Form (Figure 3). All entries must be in black ink and coincide with specimen/sample information on the Sample Identification Labels and Field Record Forms.
22. Retain one copy of the Chain-of-Custody Form and Field Record Form, place and seal all other copies in a waterproof bag, and enclose the sealed forms in the shipping container (ice chest).
23. Pack each shipping container (completely) with dry ice, secure each container with packaging tape, and seal it (e.g., across the latch of the ice chest) with a Chain-of-Custody Label. Include the signature of the sampler and the date/time sealed (in black ink) on each Chain-of-Custody Label.
24. Ship each container to the laboratory via priority overnight express delivery service, as directed by the USEPA Project Manager or Tetra Tech Task Leader. Monitor sample holding time, and factor time required for shipment/delivery to ensure that the preservation and holding criteria described in Step 18 have been met.

Figure 1. Field record for fish samples.

<b>Field Record for a PPCP Fish Tissue Pilot Project</b>				
Composite Sample ID: _____ <div style="display: flex; justify-content: space-around; font-size: small;"> <span>(State)</span> <span>(Year)</span> <span>(Site ID Name)</span> <span>(Composite # 1-6)</span> <span>(Tissue Type)</span> </div>				
Sampling Date : _____ Collection Method: _____				
Collector Name (print and sign): _____				
Affiliation: _____ Phone: _____				
Address: _____				
<div style="display: flex; justify-content: space-between;"> <div> <b>Site Location</b>  Latitude 1: _____ Longitude 1: _____  Latitude 2: _____ Longitude 2: _____  Facility Name: _____  Receiving Waterbody Name: _____  Site Description: _____  Estimated average stream depth _____ meters;    Estimated average stream width _____ meters  Stream flow (3 measurements)    _____ m/sec    _____ m/sec    _____ m/sec  Average stream flow (based on 3 measurements)    _____ m/sec </div> <div> County: _____ </div> </div>				
<b>Sample Description</b> Fish Species: _____ Total Number of Individuals: _____				
Specimen #	Length (mm)*	Weight (g)	Location	Time (Record time & time zone code below)
01	_____	_____	_____	_____
02	_____	_____	_____	_____
03	_____	_____	_____	_____
04	_____	_____	_____	_____
05	_____	_____	_____	_____
06	_____	_____	_____	_____
<b>Notes and Comments:</b> <div style="float: right; border: 1px solid black; padding: 5px; font-size: x-small;"> EST Eastern Standard Time  EDT Eastern Daylight Time  CST Central Standard Time  CDT Central Daylight Time  MST Mountain Standard Time  MDT Mountain Daylight Time  PST Pacific Standard Time  PDT Pacific Daylight Time </div>				
*minimum individual size should be no less than 75% maximum individual size				
<b>*Include Sampling Site Diagram on back of form</b>				

FORM DISTRIBUTION: White -- Tetra Tech Task Leader    Yellow -- Laboratory    Pink -- Sampler

Figure 2. Sample identification label <sup>(a)</sup>.The image shows a sample identification label template. It is a rectangular label with a notched left side. On the left side, there is a circular area with a cross-hatch pattern, likely for a hole punch. To the right of this is a table with four rows and two columns. The rows are labeled: Project Name, Site Identification, Specimen #, and Composite Sample ID # in the left column; and Date, Time, Preservative, and Collected by in the right column.

Project Name	Date
Site Identification	Time
Specimen #	Preservative
Composite Sample ID #	Collected by

Figure 3. Chain-of-Custody Form

**CHAIN-OF-CUSTODY RECORD**

Reproduced electronically

Project Manager or Client Contact:					Type of Analyses Requested										<b>Shaded area for Tt use only:</b>	
Address/Phone:			Preservative (Y/N)	Number of Containers											Sample check-in: DO _____ pH _____ Cond/Salinity _____ Chlorine _____ Appearance _____ _____ _____	
Collector Name/Phone:																
Project Number:		Project Name:														
Page	of	Sample Location:														
Date	Time	Sample Identification/Station												Collection Method	Log Number	
Sampled by: (signature)		Date/Time:		Relinquished by: (signature)		Date/Time:		Received by: (signature)				Date/Time:				
Relinquished by: (signature)		Date/Time:		Received by: (signature)		Date/Time:		Received by: (signature)				Date/Time:				

## **Appendix C**

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### **Standard Operating Procedure: Flow Measurement Procedures for a PPCP Fish Tissue Pilot Project**

## Standard Operating Procedure

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# Flow Measurement Procedures for a PPCP Fish Tissue Pilot Project

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**Scope and Applicability:** This Standard Operating Procedure (SOP) must be followed by all Field Sample Collection Teams involved with the USEPA Office of Water's PPCP Fish Tissue Pilot Project. Adherence to the SOP will ensure that field stream flow measurements will be performed the same way every time, i.e., are standardized, for all sampling participants.

Procedures for field stream flow measurement are presented as sequential steps in the SOP to follow, and include equipment, materials, and methods required to perform field measurements only.

**Responsibility and Personnel Qualifications:** This procedure may be used by any Field Sampling Teams that have been authorized by the USEPA Project Manager to collect fish for the PPCP Fish Tissue Pilot Project.

**References:** Swoffer Instruments, Inc. *Model 2100 Sensor Calibration/Care-Quick Sheet*, [http://www.swoffer.com/pdf/2100pdf/2100\\_cal\\_care.pdf](http://www.swoffer.com/pdf/2100pdf/2100_cal_care.pdf)

USEPA. 2004. *Wadeable Stream Assessment: Field Operations Manual*. pgs. 55-56. EPA841-B-04-004. U.S. Environmental Protection Agency, Office of Water and Office of Research and Development, Washington, DC.

**Precautions:** Follow usual safety precautions for working in the field. Boats should only be operated by qualified, experienced operators trained in their proper use. Each vessel must be equipped with the appropriate Coast Guard-required safety equipment (including personal floatation devices for each field team member).

### Equipment/Materials:

Sampling vessel (including boat, motor, trailer, oars, gas, and all required safety equipment)<sup>(a)</sup>

Coast Guard-approved personal floatation devices

Maps of target stream or river segments and access routes

Field Record Forms

Clipboard

Flow meter <sup>(a)</sup>

Jeweler's screwdriver

Calibration log book<sup>(a)</sup>

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<sup>(a)</sup> Selection and exact specifications at the discretion of the experienced on-site fisheries biologist.

## Procedures:

### GENERAL

Procedures provided herein focus on flow meter calibration and measurement. Field teams are also urged to read, understand, and follow the manufacturer's instructions.

### CALIBRATION

Calibration procedures presented here are for the Swoffer 2100 model flow meter (taken directly from the *Model 2100 Sensor Calibration/Care-Quick Sheet*, Swoffer Instruments, Inc., [http://www.swoffer.com/pdf/2100pdf/2100\\_cal\\_care.pdf](http://www.swoffer.com/pdf/2100pdf/2100_cal_care.pdf) )

1. Rotate the selector switch to the CALIBRATE position. A figure will appear in the display and will be either the FEET calibration number or the METERS calibration number depending on the position of the FEET/METERS switch (located in the battery compartment). For most measuring applications the calibration numbers will be about:  
FEET = 186  
METERS = 610  
If the displayed figures are much lower than these figures, the first thing to check is the battery. A weak battery can allow the indicator calibration numbers to "drift" downward slightly and will cause errors in measurements. Be sure to connect the sensor to the indicator when confirming battery strength. Always keep a full charge 9-volt battery in the compartment as a spare.
2. Mark a straight course of 10 to 20 feet in length in a body of calm, current-free water along which the sensor can be towed by walking the course. A swimming pool or dock into a quiet lake serves well. Rotate the selector switch to the COUNT position. If the display does not show all zeros, press and release RESET. (The decimal point does not show in the count mode.)
3. Place the sensor in the water a few feet before the beginning of the course, 6 to 12 inches below the surface. Make sure that the wading wand remains vertical throughout the distance traveled and that the tip of the propeller rotor faces directly into the direction of travel. Do not "crab" the rotor in the stream as you walk.
4. Begin walking the sensor through the course at a rate close to that which you will be measuring. If shallow flows are to be encountered, try to duplicate those conditions when making calibration checks. Using the wand rather than the sensor as a guide, press and release RESET at the instant the wand enters the course. The indicator will begin counting the number of sensor pulses generated as you walk. At the instant the wand leaves the course press and release START/STOP. The display now shows (and will hold) the number of pulses generated over the course length. Several passes (e.g., 2-3) through the course in both directions are recommended to develop a reliable average figure. Repeat the above process as many times as necessary to establish an average for all rotors (e.g., primary and backup).

5. Determine the average number of pulses generated through the course. If your course length is not 10 feet in length compute the number of pulses that the sensor would generate if the course were exactly 10 feet. This will be the CALIBRATION NUMBER that the Model 2100 Indicator should hold for accurate measurements with that rotor assembly in feet per second:

$$\text{FEET CAL. No.} = 10 \times \text{AVERAGE No. OF PULSES}$$

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COURSE LENGTH (IN FEET)

This number can then be multiplied by 3.281 (the number of feet in one meter) to determine the calibration number for meters.

6. Next, rotate the selector switch to the CALIBRATE position. Put the FEET/METERS switch (in battery compartment) in the "F" (FEET) position and the indicator will display the Calibration Number it presently holds for measuring in Feet Per Second. With a good battery it should be 180-186 (2" size props only). If your derived Calibration Number is different from the number displayed you can change the calibration number by using the CAL ADJUST screws at the bottom end of the indicator. Remove the CAL ADJUST cover screws (black plastic fillister-head screws). Then USING ONLY A JEWELER'S SCREWDRIVER (to prevent damage to the adjustment screw) rotate the screw clockwise to increase the displayed number and counterclockwise to decrease the number. Do the same for the Meters calibration number if necessary.
7. Each calibration adjustment screw is a 15-turn potentiometer with very fine resolution and plenty of latitude for normal adjustment given a full charge 9-volt battery. Replace the adjustment cover screws after making calibration corrections. The model 2100 indicator is not water resistant without these cover screws in place!
8. Note and store with the Model 2100 Indicator your new Calibration Number(s). Before each flow meter use, the calibration numbers and rotor assembly(s) that generated them should be confirmed and matched (rotate 2100 meter switch to CALIBRATE) before relying on readings. Also, be sure to check the calibration number while the sensor is connected to the indicator to achieve maximum battery current draw. IMPORTANT: Errors in measurements due to Calibration Number variation will be in direct percentage proportion to the difference between the ideal (correct) Calibration Number and the number that the indicator displays.

## DEPLOYMENT AND SAMPLE MEASUREMENT

1. Extend the Guide Rod slightly and swivel the Sensor to the 90° reading position.
2. Remove the Sensor Optics Protection Cap and install the Rotor Assembly on the Sensor  
*Make sure that the Set Screw is snug against the rotor shaft but do not tighten it too much to avoid stripping the threads or cracking the sensor.* Check that the Rotor Assembly spins very freely.
3. Connect the sensor to the indicator via the twist-lock connector. The connector is “keyed” and only mates one way.
4. Place the Sensor Wand vertically in the stream and point the propeller rotor into the stream flow.
5. With the Foot of the Guide Rod at the stream bed, adjust the Depth Rod up or down until the tip of the propeller is intersected by the stream surface
6. Read the stream depth on the Depth Rod Scale. Depth is measured at the top of the Slide Index fitting.
7. Next, lower the sensor until the top of the Slide Index fitting is opposite the corresponding numerical depth reading on the 6/10 Depth Scale. The sensor propeller is now at 6/10 of the stream depth from the surface of the stream.
8. Locate a cross-section of the stream channel for discharge determination that has most of the following qualities:
  - Segment of stream is straight
  - Depths mostly greater than 15 centimeters, and velocities mostly greater than 0.15 meters/second. Do not measure discharge in a pool.
  - "U" shaped cross-section, with a uniform streambed free of large boulders, woody debris or brush, and dense aquatic vegetation.
  - Flow is relatively uniform, with no eddies, backwaters, or excessive turbulence.
9. Place the wading rod in the stream and adjust the position of the probe on the wading rod so it is at 0.6 of the measured depth below the surface of the water. Stand downstream of the probe or propeller to avoid disrupting the stream flow. Wait 20 seconds to allow the meter to equilibrate, then measure the velocity. Take 3 measurements within this cross section of the stream. Record the three readings (in m/sec) on the Field Record Form. Calculate an average value (i.e., divide the sum of the values by three) and record the result on the Field Record Form.

# **Appendix D**

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## **Field Data Element Dictionary**

**Field Data Element Definitions and Instructions**  
Associated with Field Sample Collection Activities for PPCP Fish Tissue Pilot Project

Element

<b>Affiliation:</b>	The <b>Affiliation</b> field (on the Field Record Form) contains the agency, group, or company name of those persons conducting the field sampling.
<b>Collected by:</b>	The <b>Collected by</b> field on the Sample Identification Label is synonymous with the <b>Collector Name</b> field on the Field Record Form, and contains the name of the Field Team Leader.
<b>Collection Method:</b>	The <b>Collection Method</b> field on the Field Record Form contains the listing of sampling gear used to collect samples.
<b>Collector Name:</b>	The <b>Collector Name</b> field on the Field Record Form is synonymous with the <b>Collected by</b> field on the Sample Identification Label, and contains the name of the Field Team Leader.
<b>Composite Sample ID:</b>	The <b>Composite Sample ID</b> field on the Field Record Form and the Sample Identification Label is composed of a nine-character code including state of collection (two-character abbreviation), year of collection (two-number abbreviation), site identification number (three-digit city code), composite number (1 through 6), tissue type (F for fillet or L for liver).
<b>Estimated Average Stream Depth:</b>	The <b>Estimated Average Stream Depth</b> field on the Field Record Form stores an average depth estimate in meters for the stream. Estimates will be made by the Field Team through taking several measurements throughout the sampling reach.
<b>Estimated Average Stream Width:</b>	The <b>Estimated Average Stream Width</b> field on the Field Record Form stores an average width estimate in meters for the stream. Estimates will be made by the Field Team through taking several measurements throughout the sampling reach.
<b>Facility Name:</b>	The <b>Facility Name</b> field on the Field Record Form is synonymous with the <b>Site Identification</b> field on the Sample Identification Label and contains the wastewater treatment plant (WWTP) name.
<b>Fish Species:</b>	The <b>Fish Species</b> field on the Field Record Form stores the common name of the fish retained for analysis. Scientific name entries are optional.
<b>Length:</b>	The <b>Length</b> field on the Field Record Form contains the individual Total Length (in millimeters) of each fish retained for analysis. Total length of each specimen is measured from the anterior-most part of the fish to the tip of the longest caudal finray (when the lobes of the caudal fin are depressed dorsoventrally) and recorded to the nearest mm.

<b>Location:</b>	The <b>Location</b> field on the Field Record Form stores a brief description of the area in the stream where each fish was collected.
<b>Preservative:</b>	The <b>Preservative</b> field on the Sample Identification Label stores information on how the samples were preserved for shipment, i.e., either on dry ice or frozen.
<b>Project Name:</b>	The <b>Project Name</b> field on the Sample Identification Label contains the designation “PPCP Fish Tissue Pilot Project”.
<b>Receiving Waterbody Name:</b>	The <b>Receiving Waterbody Name</b> field on the Field Record Form stores the name of the stream receiving the WWTP effluent.
<b>Sampling Date:</b>	The <b>Sampling Date</b> field on the Field Record Form and Sample Identification Label stores the numerical month/date/year (e.g., 10/02/99) of sample collection.
<b>Site Description:</b>	The <b>Site Description</b> field on the Field Record Form contains a brief written description of the location of the site (e.g., road or town landmarks) and area of stream sampled (e.g., 200 m reach downstream from WWTP).
<b>Site Identification:</b>	The <b>Site Identification</b> field on the Sample Identification Label is synonymous with the <b>Receiving Water Name</b> field on the Field Record Form, and contains the name of the stream receiving the WWTP effluent.
<b>Specimen #:</b>	The <b>Specimen #</b> field on the Field Record Form and the Sample Identification Label consists of a two-digit number beginning with specimen number 01.
<b>Stream Flow:</b>	The <b>Stream Flow</b> field on the Field Record Form stores a series of 3 measurements of current stream flow recorded as m/sec.
<b>Average Stream Flow:</b>	The <b>Average Stream Flow</b> field on the Field Record Form consists of a single (calculated) average of the 3 stream flow measurements, recorded as m/sec.
<b>Time:</b>	The <b>Time</b> field on the Sample Identification Label and Field Record Form stores time of sample collection recorded in military time (i.e., four digits). Time fields on the Field Record Form include collection times for each individual specimen.
<b>Weight:</b>	The <b>Weight</b> field on the Field Record Form stores the individual Total Weight (in grams) of each fish retained for analysis.

## **Appendix E**

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### **Resolved Action Form**

**FISH SAMPLING ACTIVITIES  
DATA REVIEW  
RESOLVED ACTION FORM**

Date: \_\_\_\_\_  
Prepared

Composite Sample ID: \_\_\_\_\_

*Attach this form to corresponding Fish Sampling Activities Data Review and Field Record Forms, and return all deliverables to the EPA Project Manager.*

Prepared by: \_\_\_\_\_ Signature: \_\_\_\_\_

Elements requiring resolution	Corrective Action	Resolution authorized by:
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

*Forward original form and attached Data Review and Field Record Forms to the EPA Project Manager.  
Copy this form and retain with Data Review and Field Record Forms in Tetra Tech Project File.*

## **Appendix F**

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### **Field Sampling Activities Data Review Form**

## FISH SAMPLING ACTIVITIES DATA REVIEW

Date: \_\_\_\_\_  
sampled
received

Collector: \_\_\_\_\_

Composite Sample ID: \_\_\_\_\_

*Return this form and the attached deliverable (on which comments or corrections  
have been written and initialed) to the EPA Project Manager.*

Reviewer: \_\_\_\_\_ Signature: \_\_\_\_\_

Date: \_\_\_\_\_  
reviewed

Review Elements	(Circle One)	If "No," describe condition and corrective action.
1. Is Composite Sample ID number entered correctly?	Yes No	_____ _____ _____
2. Does the site ID number correctly correspond with the site name (as per the target site spreadsheet)?	Yes No	_____ _____ _____
3. Are field collectors identified on the form?	Yes No	_____ _____ _____
4. Have site coordinates been entered on the form?	Yes No	_____ _____ _____
5. Is a fish species identified on the form?	Yes No	_____ _____ _____
6. Do fish lengths meet the 75% rule (shortest length divided by the longest length > 0.75)?	Yes No	_____ _____ _____
7. Are there 3-4 fish in the sample? If "No", how many fish are in the sample?	Yes No	_____ _____ _____
8. Has the Field Record Form been completed in its entirety? If "No", list missing information.	Yes No	_____ _____ _____

Additional Comments and Notes: \_\_\_\_\_

Overall Evaluation:    ☐ Acceptable    ☐ Unresolved or Needs Additional Review

*Forward original form and attached Field Record Form to the EPA Project Manager. Copy this form and retain with Field Record Forms in Tetra Tech Project File.*

## **Appendix G**

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### **Field Observation Plan**

## **PPCP Fish Tissue Pilot Project FIELD OBSERVATION PLAN**

Site Observer: USEPA or Tetra Tech QA Representative

Purpose: To observe field operations and adherence to field sampling protocols.

Each Site Observer will receive a package containing the following field sampling monitoring items:

- Field Observation Plan (1-page)
- Field Observation Checklist (3-page carbonless copy form) [2 copies]
- Field Sampling Standard Operating Procedures (May 2006)
- Sampling Site spreadsheet
- Clipboard/pen
- Stamped, pre-addressed envelope to return completed forms

Site Observers should follow the series of steps below to complete this QA/QC activity.

1. Review Field Observation Checklist with Field Sampling Team Leader. Prepare materials and equipment.
2. Arrange schedule of visitation with the Field Sampling Team Leader and confirm site location, where to meet the team, and how to get there.
3. Observe performance of the team through one complete set of sampling activities as detailed on the Field Observation Checklist.
4. Complete Field Observation Checklist, including comments for any element that is partially met. Summarize field observations and note any significant issues on the last page of the Field Observation Checklist before signing and dating the form.

Note: A significant issue is defined as the team overlooking a step in the operation, incorrectly performing a step, or taking any action that could adversely affect the chemical analysis of the fish tissue. Examples include incomplete recording of data on the Field Record Form and inappropriate handling, packaging or labeling of fish samples.

5. Retain bottom (pink) copy of the Field Observation Checklist for your records. Forward the original (white copy) and the yellow copy to the EPA Project Manager in the stamped, pre-addressed envelope.

## **Appendix H**

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### **Field Observation Checklist**

# PPCP Fish Tissue Pilot Project FIELD OBSERVATION CHECKLIST

**Date of Assessment**\_\_\_\_\_ **Site Observer**\_\_\_\_\_

**Site Name**\_\_\_\_\_ **Site ID Number** \_\_\_\_\_

**Field Team Leader/Affiliation**\_\_\_\_\_

**Note:** A check mark under *Yes* means that the requirements of the element are fully met. A “P” under *Yes* means that the requirements of the element are partially met; this must be explained under Comments. A check mark under *No* means that the element was omitted or not performed correctly.

Element Evaluated	Yes	No	Comments
<b><i>Field Sampling Activities</i></b>			
Roles and responsibilities are discussed at a pre-event briefing and are understood by each Team member.			
Team has all necessary sampling equipment, forms, sampling materials, labels, and safety equipment. (See “Table 4. Equipment and Supply Checklist for Fish Tissue Sampling” in the Sample Collection Activities QAPP for the PPCP Fish Tissue Pilot Project.)			
Site is determined to meet the following criteria: - serves as receiving waterbody for WWTP listed on Sampling Site spreadsheet - has a viable fish population of a resident species that is subject to effluent exposure for much of its life cycle and is typically consumed by wildlife and humans			
Passive collection devices (if used) are checked frequently (e.g., several times daily if possible, but at least every 24 hours) to ensure a limited time lag between fish entrapment and sample preparation.			
A total of 6 composite samples of the same species are collected, rinsed, and identified. Clean nitrile gloves are worn during sample handling.			
The selected fish are suggested target species, or an appropriate substitute species for that particular region.			
The selected specimens all satisfy legal requirements of harvestable size and weight, or are at least of consumable size if no legal harvest requirements are in effect.			

Element Evaluated	Yes	No	Comments
Composites consist of at least 3 fish of adequate size (to provide at least 30 grams of edible tissue and 10 grams of liver tissue).			
The length of each fish is measured and recorded in mm from the anterior-most part of the fish to the tip of the longest caudal finray.			
The selected specimens all are of similar size, so that the smallest individual is no less than 75% of the total length of the largest.			
The weight of each fish is measured and recorded in grams.			
The selected specimens are all collected at the same time (i.e., as close as possible, but no more than one week apart).			
Team completes, signs, and dates Field Record Form.			
<b><i>Sample Handling and Integrity</i></b>			
Each fish is wrapped in (laboratory provided) heavy-duty aluminum foil.			
Sample ID Label is prepared for each fish.			
Lengths of food-grade tubing are cut that are long enough to contain each individual fish and allow extra length on each end to secure with cable ties.			
Each foil-wrapped specimen is placed in the appropriate length of tubing. Each end of tubing is sealed with a plastic cable tie, and the appropriate Sample ID Label is attached.			
Wrapped specimens are placed inside a large plastic bag with all specimens of the composite from that site and sealed with another cable tie.			
As soon as each sample is packaged, it is placed immediately on dry or wet ice for transport to an interim facility for freezing before shipment.			

Form Distribution: White – EPA Project Manager    Yellow – Tetra Tech    Pink – Field Observer

Element Evaluated	Yes	No	Comments
Chain-of-Custody Form is completed. All entries are written in black ink and coincide with specimen/sample information on the Sample Identification Labels and Field Record Forms.			
One copy each of the Chain-of-custody Form and Field Record Form are retained by the field sampling team.			

**Summary of Observations:** (Attach additional sheets if necessary)

**Significant Issues Identified? (Circle one) Yes No**

If yes, briefly describe corrective action:

**Other Comments or Notes:**

**Signature of Site Observer:**

Signature \_\_\_\_\_ Date \_\_\_\_\_

*Form Distribution: White – EPA Project Manager Yellow – Tetra Tech Pink – Field Observer*